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Proceedings of THE CONFERENCE ON CONTINUUM ASPECTS OF GRAPHITE DESIGN

November 9-12, 1970
Gatlinburg, Tennessee

United States
Atomic Energy Commission

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Edited by W. L. Greenstreet and G. C. Battle, Jr.

Held under the auspices of the
AEC Graphite Coordination Working Group

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FOREWORD

The Conference on Continuum Aspects of Graphite Design was held to disseminate and exchange information from a number of endeavors, each with a unique purpose, but all germane to the design and structural analysis of graphite components. In recent years, extensive work has been done and significant advances have been made in characterizing the mechanical behaviors of graphites, in developing analytical methods for use in design analyses, and in establishing failure and design criteria. The fact that structural design technology associated with graphite components has advanced a great deal in the past decade is evident from the program of this conference. However, in the main, the work has been fragmented, in the sense that it has been conducted by diverse groups aimed toward specific, but differing, objectives. Thus, the conference was organized to focus in a unified fashion on the areas of technology applicable to design in general. Persons working in the various fields were brought together to exchange information, and to broaden the general perspective regarding necessary input and interaction for successful structural design of high performance graphite components.

The basic topics, or areas, covered were:

1. Material Characterization,
2. Constitutive Equations,
3. Structural Analysis Methods and Applications,
4. Fracture,
5. Structural Design Criteria.

Pursuits in these areas provide the essential ingredients required in design. Since the papers presented were concerned with current studies and activities, these proceedings provide a basis for assessing the current state of the art. Additional topics covered were thermal shock, ablation, and other special topics of general interest.

Oak Ridge National Laboratory's interest in the continuum aspects of graphite design had its genesis about 1960 when it became apparent that available data and design methods were not adequate to allow assessment of the structural adequacy of the graphite moderator and reflector elements for the Experimental Gas-Cooled Reactor (EGCR), which was built at Oak Ridge by the U.S. Atomic Energy Commission. An intensive

study of the related areas was initiated at that time. Since then, the interest and activities have been broadened to include work on components for nuclear reactors for space vehicle propulsion as well as molten-salt and high-temperature gas-cooled reactors for central power station use. Because of this background, we, in the Applied Mechanics Section of the Reactor Division, were very much interested in holding a meeting of this type and were pleased with both the opportunity and the timing.

The Conference was held by Oak Ridge National Laboratory under the auspices of the Graphite Coordination Working Group. The Working Group was established by the U.S. Atomic Energy Commission for the purpose of reviewing and assessing work on graphite technology development. The subjects of this conference are therefore important parts of the overall sphere of interest of this Group. The cooperation and encouragement of USAEC and NASA groups as well as the participation and interest on the part of the USAEC, the U.S. Air Force, NASA, and private industrial organizations were especially beneficial in making this conference possible.

The papers and discussions incorporated in the Proceedings were edited only to the extent considered necessary for the reader's assistance. The views expressed and the general style adopted remain the responsibilities of the named authors or participants. The affiliations of authors and participants are those given at the time of the meeting.

These Proceedings should be of general engineering interest as well as of specific interest to those working directly with graphites. Since the present state of the art is represented here, it should be very useful to those in the latter category.

W. L. Greenstreet
Conference Chairman

ACKNOWLEDGMENTS

The efforts of the many individuals who contributed to this conference are gratefully acknowledged. Special thanks go to the authors; to the conference coordinators, J. P. Callahan, G. T. Yahr, and R. S. Valachovic; to the session organizers and chairmen (the names of these individuals appear elsewhere in the proceedings); and to the ORNL Conference Staff, C. E. Normand, coordinator. Sincere appreciation is also extended to F. L. Culler, Deputy Director of ORNL, and W. S. Scheib, Division of Space Nuclear Systems, USAEC, for their encouragement.

W. L. Greenstreet
Conference Chairman

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INTRODUCTORY REMARKS

J. E. Fox, USAEC

Co-Chairman, Graphite Coordination
Working Group

On behalf of the Atomic Energy Commission, I'd like to welcome each of you to this conference.

One of the objectives of the meeting will be to have everyone go away knowing what the title meant! As the announcements have indicated, the conference is being held under the auspices of the Graphite Coordination Working Group. I'd like to make it very clear that all of the work involved in organizing the meeting has been done by Bill Greenstreet and his associates at the Laboratory, and also the session organizers. All the Working Group really did was to agree with Bill's submission that a meeting on this subject was needed, and that ORNL would be an ideal sponsor for the meeting.

The Graphite Coordination Working Group was organized in January 1967. Its members include five AEC representatives and 11 contractor representatives. The contractors currently include Battelle Northwest, Gulf General Atomic, Los Alamos, Oak Ridge National Laboratory, Penn State University, Sandia, Westinghouse, and the Y-12 Plant. The general objective of the Working Group is to improve coordination between the various Graphite R&D programs sponsored by different divisions of the Atomic Energy Commission and by other Government agencies.

This meeting is the fifth to be held so far under the auspices of the Working Group. It is the second to include a significant number of papers on non-AEC-sponsored work. This non-AEC work is predominantly work supported by Air Force DOD contracts, but also includes, as you will see today, work privately funded by graphite suppliers.

I think you will agree with me by the end of the meeting that this conference is a good example of an important function of the Working Group, and Bill has done an excellent job in organizing the conference.

OAK RIDGE NATIONAL LABORATORY AND ITS GRAPHITE PROGRAMS

Opening Address

D. B. Trauger

Associate Director for Engineering Programs
Oak Ridge National Laboratory

On behalf of the Oak Ridge National Laboratory I wish to welcome you to this meeting on the Continuum Aspects of Graphite Design - whatever that means. I am reminded of a staff member in our Metals and Ceramics Division, a welding expert, who commented on graphite, somewhat in frustration: "It is not a metal; it is not a ceramic; why, it's only a chemical!"

In reviewing the paper titles for this meeting I think you may have a still different impression of graphite. It could be a structural analyst's dream or nightmare, as the point of view may be. Graphite seems to offer, by its complexity, an infinitum of variety for study. Perhaps it is a little like East Tennessee golf courses, which offer great variety but also produce certain frustrations.

Regardless of the viewpoint, the program for this meeting represents work of great interest and I am pleased with the privilege to hear some of the papers today. Rather than discuss graphite in the context of your meeting, I will comment on those aspects of ORNL which relate to our energy programs and emphasize those projects which involve graphite.

As you know, one of the earliest applications of graphite in the nuclear field was for the Chicago Pile. The next step was the construction at X-10, later to become the Oak Ridge National Laboratory, of the first operating reactor. It was built basically of nuclear-grade graphite in a 24-foot cube which contained the aluminum-clad uranium fuel slugs and was cooled with air discharged through a stack. After being constructed in a record period of six months and operating for 20 years, the reactor was retired and appropriately made a National Historic Landmark.

Out of this beginning ORNL developed as a Laboratory with great interest in the production of energy, particularly for converter and breeder reactors, and in the chemical processes for the recovery of plutonium, the

separation of fission products, and the production of radioisotopes. From these tasks have grown wide interests in the secondary effects of nuclear energy programs including safety, radioactive waste disposal, and ecological and biological effects.

Figure 1 shows some aspects of the Laboratory when viewed with respect to its interests in the production of energy.

One of our major programs has been the development of breeder reactors which operate in the thermal neutron energy region, and which utilize thorium as the primary fuel source. The ORNL Molten Salt Breeder Reactor has a liquid fuel in the form of uranium tetrafluoride dissolved in a carrier salt primarily of lithium and beryllium fluorides. The flow schematic of Fig. 2 shows the electric power generating train at the right and a simplified chemical processing circuit at the left. The latter removes protactinium and fission products having high neutron cross sections. Gaseous products such as xenon-135 are removed by a sparging system.

An important advantage of this reactor system is the combination of power production and fuel reprocessing which avoids the need for shipping highly radioactive fuels to reprocessing plants. Doubling times for fuel on the order of 15-20 years can be achieved.

The reactor uses graphite to adjust and optimize the neutron energy spectrum. Figure 3 is a cutaway view for a reactor vessel with the graphite assembly. This is the Molten Salt Reactor Experiment, which operated at approximately 8 MW with excellent availability over a four-year period.

Figure 4 shows the graphite during assembly and Fig. 5 shows one graphite unit.

There are special requirements for the graphite. The fuel salt does not wet the graphite and tends not to enter the pores. However, it also is necessary to keep Xe-135 from entering the pores and thereby remaining in the reactor as a poison. Thus the graphite or its surface must have a very low permeability. It also must have a high tolerance for fast neutron damage, and development programs are in progress to improve the performance in each of these respects. Graphite now available has a projected lifetime for a breeder of perhaps four years, at which time it would be replaced, but for a converter reactor with a lower power density it is now possible

to project a 30-year lifetime. Conceptual designs are being developed for prototype reactors.

Since 1957 we have been studying and developing the technology for gas-cooled reactors using helium as the coolant. The most advanced design now uses graphite as the moderator and for the fuel element structure. Figure 6 is a cutaway view of a large HTGR by Gulf General Atomic.

The fuel element is a rather complex block of graphite which contains coated particles, as shown in Fig. 7. The basic fuel unit is a small kernel, perhaps 200 to 600 μ in diameter, of the oxides or carbides of thorium and uranium. The particles are coated with pyrolytic carbon, and in some cases silicon carbide, to provide a gas-tight enclosure. All particles have multiple layers. The inner layer serves to absorb fission product recoil energy and is porous to provide space to accommodate gaseous fission products and fuel swelling. These particles are highly developed and can sustain fuel burnups greater than 50 atom % and fast neutron fluences of 8×10^{21} nvt at temperatures in excess of 1250°C.

In Fig. 8 we see a coated particle which has been heat treated at a very high temperature, approximately 2500°C. We normally think of pyrolytic carbon as a brittle material. However, this coating under the internal pressure from the reaction of the oxide fuel with the pyrolytic carbon to form CO has expanded like a rubber balloon.

An important ORNL role in the development of this reactor has been the design and performance under irradiation of the coated particles and graphite. We have also studied the resistance of the graphite to oxidation by steam which might enter the helium circuit from steam generator leakage. A major role at the present time is the development of a remote recycle pilot plant to demonstrate the recovery and refabrication of U-233 as the fuel.

Although many possibilities remain for the improvement of HTGR fuel, the reactor is now ready for commercial application. Four small prototypes have been operated for several years. A 330-MWe prototype reactor is now approximately 70% complete near Denver, Colorado, and others are planned in Europe. Both the HTGR and the MSR can deliver modern steam conditions at thermal efficiencies of $> 40\%$, thus eliminating the disadvantage of nuclear power stations in dissipating waste heat to the environment. The

HTGR also is suitable for a direct helium turbine cycle. In this case it is desirable, although not necessary, to further improve the graphite and fuel particle coatings to obtain still higher temperatures.

Finally, I would mention some work for space nuclear rocket applications. We are extremely fortunate in having the major development effort on NERVA graphitic fuel elements at our sister organization in Oak Ridge, the development division of the Y-12 Plant. They have supplied us with experimental graphites for irradiation testing in the HFIR. Since these materials are formed from known raw materials and with carefully controlled processing techniques, the irradiation effects can be traced directly to the variations in the basic materials. We think that these will play an important role in the development of graphite, not only for the Space Program, but also for the molten salt reactor and to a lesser extent for the HTGR. Irradiation testing for these programs provides important information relative to the basic nature of the graphite.

NERVA work, of course, spans a very wide temperature range from cryogenic to greater than 5000°F. Mechanical behavior studies on Rover and other graphites have been made at room temperature, and studies of commercial graphites have been conducted at the maximum temperatures of interest. Currently, three types of studies are under way in a continuation of graphite mechanics work. These are:

- (1) examination of failure criteria in order to predict modes of failure for graphite components,
- (2) study of the mechanical behavior of fuel and central support elements at high temperature,
- (3) obtaining thermal rupture resistance measurements on specimens of fueled and unfueled materials for the NERVA program.

In each of these studies, the development of mathematical characterizations is emphasized. Test programs include short time behavior, creep, and creep recovery tests for both uniaxial tensile and compressive loadings, relaxation tests for tensile loading, and dynamic modulus tests. In addition, we expect to set up and calibrate test systems to conduct thermal shock tests on NERVA fuel materials as well as other mechanical properties

and to measure the thermal conductivity of graphites at cryogenic temperatures.

To go further I would be intruding into the topics of your meeting. As you will note, much of the Space Program-related work is being reported in detail and there are papers on the Molten Salt Reactor graphite development and properties of pyrolytic-carbon-coated particles.

It seems evident from these few illustrations that graphite can serve for a multitude of purposes. With the substantial development programs under way to produce new and improved materials of carbon, I predict that there will be a continuing need for and great value in your studies which are basic to the understanding of this important material. The interchange of information which this meeting provides should be a most valuable contribution to the continuation of vital programs at each laboratory which you represent. We of course hope that it does fulfill this expectation and wish to do all that is possible to make your visit to East Tennessee enjoyable and worth while.

Fig. 1. Nuclear Energy Programs at Oak Ridge National Laboratory.

Fig. 2. Flow Schematic for the ORNL Molten Salt Breeder Reactor.

Fig. 3. Reactor Vessel for the Molten Salt Reactor Experiment, Showing Graphite Assembly.

Fig. 4. MSRE Graphite Assembly.

Fig. 5. A Single Graphite Unit for the MSRE Graphite Assembly.

Fig. 6. Large HTGR General Arrangement by Gulf General Atomic.

Fig. 7. HTGR Coated Fuel Particle and Fuel Element.

Fig. 8. Comparison of Microradiographs of Coated UO_2 Particle Before and After Heat Treatment for 10 min at 2500°C.

DESCRIPTION OF DESIGN REQUIREMENTS FOR GRAPHITE COMPONENTS*

Keynote Lecture

J. C. Rowley
Los Alamos Scientific Laboratory
Los Alamos, New Mexico

ABSTRACT

The requirements to be described include the broad prospects for utilization of graphite in the design of high-temperature gas-cooled and molten-salt reactors for power plant use, nuclear rocket reactors, and reentry nose cones and heat shields.

Some personal observations about the progress and trends in graphite technology and utilization in structural design and applications over the past decade will be made. The major residual problems for the application of graphites for both exacting short term service and long time reactor operations appear to center around: initial selection and design efforts often based on insufficient familiarity with what finally turn out to be the most important material characteristics; a general inability to design with an inherently brittle material; lack of sufficient recognition of low strength (compared with most technologically important metals) of graphites; improper account for peak mechanical loads and attachment restraints; failure to recognize the need for very accurate analytical stress analysis results and definition of significant extreme operational loads or thermal gradients; and a lack of good design practices relative to stress concentrations and the statistical aspects of strength reductions of large volume components relative to small-specimen strength test data. Finally, there is often a failure to recognize the need for accurate descriptions of thermal and mechanical boundary conditions if adequate structures and optimum designs are to be achieved.

Graphite technology still lacks a comprehensive and practical thermal-mechanical continuum model to account for important inelastic effects for both short-time loads and long-term creep deformations. In addition, the significant parameters needed to define fracture criteria have yet to be formulated. In particular, graphite is often selected for its excellent thermal-stress fracture resistance; however, few quantitative results in this area are available,

*Work performed under the auspices of the U.S. Atomic Energy Commission.

and this field of endeavor is particularly hampered by the lack of realistic fracture criteria and applicable continuum models noted above. Recent results at ORNL and elsewhere indicate that we will be able to start to move away from the current need to apply linear elasticity and a maximum normal stress criterion of failure and from the attendant necessity of the use of large "safety factors" and expensive component test programs.

Graphite technology and design applications will benefit from progress in the general field of brittle materials. There are currently some trends toward more understanding of the problems encountered in this area. However, as in the case of graphite technology advances, there are rather widely dispersed efforts, and much of our emphasis remains on the attempts to obtain solutions to problems for a specific application. Perhaps the encouragement provided by such meetings as this will help avoid some of the present duplication of efforts, encourage more interchange of knowledge, and alleviate the present fractionization of views on graphite structural designs and applications.

I. INTRODUCTION

There are currently in the United States, and abroad, a rather large number of advanced technology programs which are based upon the selection of various graphites as a structural material. These selections were made with the very unique properties of graphite in mind. Clearly of major importance in these selections is the ability of graphite material to perform a structural function at very high temperatures in extremely hostile environments. Often no metals or alloys are available which can approach the performance of graphite for the intended demanding applications. In high-temperature nuclear reactor applications particularly, the nearly unique combination of neutron moderation-reflection capability and extreme refractory properties of graphite is often dominant in the choice.

In addition, the relatively high thermal conductivity of the polycrystalline graphites allows high internal energy generation rates (e.g., high reactor power density) to be extracted without excessive temperature gradients and attendant thermal stresses or large thermal-strain distortions. Thus, attractive reactor fuel element designs are possible, and non-fueled graphite reactor structures which receive considerable radiation

heating can be devised which look very attractive. The high heat conductivity is also aided in reducing thermal-stress and strain levels by the low thermal expansivity and modulus exhibited by the majority of commercial polycrystalline graphites. Such a combination of properties also makes graphite an attractive material choice for applications requiring the tolerance of large and rapidly applied heat fluxes for such structures as nose cones and ablation tips for atmospheric reentry vehicles.

In contrast to the situation a decade ago, there now exists a wide range of literature which can serve as a general basis for study and background information for newcomers in the field of structural applications of graphite. One might just mention a few of the available sources to indicate the breadth of literature in the field: Mantell's Carbon and Graphite Handbook¹; the excellent journal, Carbon²; Nightingale's book, Nuclear Graphite³; the series edited by Walker, Chemistry and Physics of Carbon⁴; from Britain, Reynold's recent survey, Physical Properties of Graphites⁵; and for those whose French is passable, Les Carbones⁶; and finally, of course, the published proceedings of the Carbon Conferences. For some more specialized fields, there have been developed good sources: The ASTM's Nondestructive Testing of Nuclear Graphite⁷; and for nuclear reactor applications of graphite, the AEC's journal Reactor Technology⁸, which attempts to keep up with the report literature and often contains surveys of interest to reactor graphite applications. For a person specifically concerned with mechanical design and mechanical properties behavior, general information is very limited, and the effort of Greenstreet's group here at ORNL is probably the most notable (as represented by a recent ORNL report⁹).

Finally, one should mention the recent useful AFML guide¹⁰ to commercial sources and grades of graphites, which provides a quick entrée into what is generally available and provides a broad scoping of graphite properties.

Certainly, the information situation is vastly improved over the nearly complete reliance we had to place on the materials volume of the Reactor Handbook in the initial phases of our Rover Program design efforts.

It is probably true that much of the present interest in graphite structures and applications is a result of confidence gained from the successful applications in several major national programs. Specifically, one should mention, of course, our Rover Nuclear Propulsion Program at Los Alamos¹¹ and the derived NERVA (nuclear rocket engine) program, Fig. 1; also Los Alamos' power reactor experiment, UHTREX,¹² Fig. 2 (now placed in "moth-balls"); and the Gulf General Atomic power reactor program which produced the HTGR-Unit 1 (Ref. 13). The several successful European reactors and reactor design efforts based on graphite could also be cited.

Much of the growing importance of graphite in high-temperature advanced technology applications is due to advances made in materials technology, as pointed out in the 1967 review by Goeddel and Siltanen.¹⁴ However, it should be pointed out that mechanical design and stress analysis techniques for graphite structures have not kept pace with other areas of graphite knowledge. As witness to this view, one would cite the continued widespread applications of linear elastic (continuum) behavior, classical plasticity models, and metal-like creep models in many graphite structural analyses.

II. CURRENT GRAPHITE APPLICATIONS AND DESIGNS

1. Nuclear Power Reactor Applications

A number of high-temperature nuclear reactor system designs are being developed to meet the world's future civilian power needs. Rather extensive research and design efforts have been initiated both in the United States and abroad. The urgency and nature of these coming power crises and basis for the reactor programs have been clearly outlined in a recent National Academy of Science report.¹⁵

The interest in high-temperature systems and, therefore, the applications of graphite structures in such systems is obviously based on achieving increased thermal efficiency of the proposed plants. Also, the choice of graphite is strongly weighted by the excellent and well-known neutronic properties of graphite. The attainment of future economic power and the required fuel breeding in these plants will obviously be intimately connected to the developments and improvements in graphite

technology. A brief review of two of the U.S. programs: The High-Temperature Gas-Cooled Reactor (HTGR) by AEC-Gulf General Atomic and the Molten Salt Breeder Reactor (MSBR) program by AEC-ORNL will indicate the sizes of these and similar ambitious programs.

A photograph, Fig. 3, of the HTGR core being designed for the Public Service Company of Colorado illustrates the size of such systems and the amount of graphite structures involved. Table 1 gives parameters for the plant and illustrates the use of graphite in the fuel elements: cylinders 20 ft long and 4.5 in. in diameter, 2000 per core, operating at $\sim 1260^{\circ}\text{C}$; and the use of graphite as moderator. A review¹⁶ of current stress analysis problems of such core-fuel assemblies indicates the need to predict fuel bowing and graphite creep stresses and strains over a six-year core life, under doses of $\sim 6 \times 10^{21}$ NVT. Much of the stress and deformation analyses are based upon linear elastic and linear viscoelastic behavior assumed for the graphite. Typically, what one finds is that excessively high stresses may be predicted in the fuel at the end of four years, that the uncertainties are apparently largely due to the unknown relation between the actual behavior of graphite in the reactor environment and that assumed as the model for the graphite behavior in the analyses.

A second illustration is the Molten Salt Breeder Reactor (MSBR) program, Fig. 4. Parameters influencing the graphite components are given in Table 2. The importance of graphite in the design and performance of this reactor system is indicated in a recent ORNL report,¹⁷ where a very preliminary estimate of the irradiated-induced stresses in the MSBR is given. The analysis considers differential distortions caused by temperature and flux gradients within the graphite core tubes or "cells". The dimensional changes are estimated from an empirical relation which depends on flux and local temperature (~ 550 to 750°C). Creep strains are then calculated for a circular tube geometry. The important item for us to note, is that the creep response (model) for graphite appears to be assumed as a constant volume process (i.e., Poisson's ratios are taken to be $1/2$), a circumstance known to be a good approximation for metals, but not thought to be valid for graphites.¹⁸ Indeed, quite the reverse appears to be the case, since graphites appear

to deform with essentially constant values of Poisson's ratio (perhaps more properly, strain ratios). Also, changes in density occur with little or no "necking down" observed in uniaxial tensile (or creep) tests. Further, preliminary stress values are calculated from this assumed continuum model for the behavior of the graphite to be utilized in the reactor core. They are said to be of the order of 10% of the expected "strength" which might be expected for the proposed graphite. No mention is made of the type of strength measurements or of any size effects which might be expected to reduce the "strength" of large blocks below those of smaller test specimens.

In view of the impact of such graphite stress analyses on the predicted reactor lifetimes and consequent important economic implications, it would seem that more attention to the development of more correct and appropriate continuum models for graphite mechanical behavior would be actively sought.

2. Reentry Applications

The selection of graphites for applications to reentry nose cone and nose tip structures appears to be primarily based upon its legendary thermal shock resistance. A typical graphite nose cone structure is shown in Fig. 5. The major source of stress in such structures is generated by the very rapidly encountered large heat flux which occurs at the surface of the nose cone during reentry into the atmosphere, Fig. 6. Aerodynamic pressure loads and ablation phenomena also need to be considered in the design of these structures.

The major design consideration for such graphite structures is thermal shock, or rapidly applied transient temperature gradients. The usual analytical approach to stress analysis of such structures¹⁹ assumes the graphite to be a linear elastic body (often with great care taken in accounting for the appropriate anisotropy). Alternatively, the approach is often a classical elastic-plastic analysis which implies that the plastic or nonrecoverable volume dilatation is zero, whereas test data at both room temperature²⁰ and elevated temperatures²¹ have shown that appreciable permanent dilatation does occur.

Moore²² has attempted to assess the differences in stress and strains originating from such assumptions, for a relatively simple, room temperature graphite structure: a thick-walled cylinder with internal pressure. Figure 7 shows these stress analysis results, and indicates the differences in models. More important perhaps, was the excellent agreement achieved by this permanent-dilatation model with the strain gage data for the outside surface. It seems quite obvious that more accurate predictions of graphite thermal stresses (and strains) and irradiation-induced creep behavior could be achieved by this more accurate continuum model, since these processes are essentially dilatational in nature.

3. In Summary

It seems obvious that the use of more realistic constitutive relations (continuum models) will allow structural designs with graphite to take advantage more fully of the strength of graphite, low though it may be. Continued effort to develop both models and the necessary supporting experimental data for structural graphites will surely produce more optimally designed graphite structures.

III. GRAPHITE PROPERTIES AND DESIGN

The structural analysis of graphite components is nearly always hampered by insufficient properties data for the specific grade under consideration. This lack often stifles initiative in adopting the more accurate and complex continuum models which are available to describe graphite behavior. While it is often possible by conservative and clever design approaches to overcome these areas of ignorance, it is obvious that system optimizations will require increasingly accurate descriptions of the material behavior.

The most prominent difficulty is the inherently brittle nature* of graphite. An error in design, or analysis, an unexpected or

*This is perhaps an overstatement of the case. There may indeed be found elevated temperature and hydrostatic pressure states where plastic flow (crystallite slip) will actually be observed.

unaccounted-for high mechanical loading, a larger than expected thermal gradient applied to the graphite component will inevitably lead to cracking or fracture. There is no recourse to a little ductility helping over a small design oversight or unexpected overload condition. Thus, accurate descriptions of graphite fracture behavior become a problem in common with the design of all brittle materials.

Several excellent reviews of brittle-materials behavior have become available^{23,24,25}; however, from a practical point of view, these reviews do little more than clearly point out the problems that need to be tackled and the research directions in brittle-materials technology which are needed. It is safe to say that graphite structural design will benefit from much of the materials technology advances achieved in the understanding of brittle-materials behavior in general, and studies of brittle fracture particularly.

This leads us to consider perhaps the most needed area of research in graphite properties. The optimum design and analysis of graphite structural components is currently most seriously hampered by the lack of an accurate description of graphite fracture. Even if we do have and apply the most accurate continuum models and constitutive relations (and, of course, use completely accurate values of applied loads and restraints), structural analysis is, at present, unable to prescribe the combination (or combinations) of these very accurately derived stresses and strains which describe failure or fracture in graphite. It is rather common practice today to utilize the maximum normal component of the tensile stress component derived in the analysis. And then, usually in a rather vague manner, compare this value with some strength data (usually whatever data is at hand). Often, uniaxial tensile tests or modulus of rupture (bend test) data are evoked for such comparisons. These data are frequently developed at great cost over the intended temperature ranges of the particular applications. And the strength data may even be obtained with great attention to determining the anisotropy of the original graphite billet. In addition, strength variations within billets and from billet to billet are often sought. Indeed, great care in obtaining reliable and significant statistical sampling is often practiced. But in the end, it is a rare graphite

component whose critically stressed areas resemble the simple stress and strain state of the strength tests. Thus, the analyst and designer, for all their care, and after numerous computer runs, really are unable to make very knowledgeable predictions of the likelihood of fracture of the graphite component.

In the sense of continuum mechanics, we need additional thorough efforts in the area of the development of a fracture criterion for graphite.

I don't think it is appropriate to attempt to review further, here, the graphite fracture work that has been accomplished and the research efforts currently under way. Session 4 of this meeting should give us a chance for an up-to-date review of these matters and provide an opportunity to urge more definitive efforts in this general area.

Many other material properties enter into the analysis of a high-temperature graphite component. Their accurate determinations and evaluation are of course, important as inputs to the design analysis. However, their need is usually well recognized and values of most properties are reasonably easily obtainable, even if costly and time consuming. Most of us are also well aware that for design analyses requiring accurate results, the specific type or grade of graphite under consideration for a design must be sampled.

IV. ANALYSIS OF GRAPHITE COMPONENTS

1. The Revolution

In sharp contrast to the situation ten or even five years ago, the analyst now has a very formidable arsenal of analytical methods at his disposal to represent complex material behavior, can utilize very general geometries, and can account for distributed mechanical loads and complex temperature fields. Indeed, these analysis methods, which are usually in the form of computer programs, constitute a revolution in structural analysis. It is not an exaggeration to assert that it is now possible to account for the most complex, nonlinear, inelastic, time-and-temperature-dependent material behavior observed in nature. This can be accomplished to almost any degree of refinement and detail that a

particular application warrants (and one can afford to apply). Almost all modern and up-to-date advanced technology development groups and organizations have their stables full of such thermal, fluid-flow, heat transfer, and structural analysis computer codes. A glance at the reports of almost any aerospace, nuclear reactor, or other advanced technology program and one sees, scattered throughout them, various letter acronyms for these favored computer programs. (They are often touted by title, much like thoroughbreds in a racing stable.) The extensive, intelligent, ingenious, and rapidly growing application of the various so-called finite element methods has made much of this analytical breadth and versatility possible.

The inconsistency we now encounter is that an equivalent revolution has not yet occurred in the materials technology fields. Thus, the analyst often finds himself without the proper material behavior description to utilize in his analysis (computer programs). What may happen then is that the analyst will make up (assume) a best guess as to the general nature or character of the material behavior, or even some convenient continuum model of the material may be adopted, and then the analyst produces stress and strain results in great abundance. Often a large parametric study will result, with various geometries and material parameters inserted into the analysis, and a mass of plots or tables of results is produced. The stress magnitudes are then (often rather apologetically) compared with the rough order of magnitude of some (rather haphazardly obtained or selected) "strength" values. Far reaching design and program direction decisions are then based on such stress analysis outputs! (Indeed, a paper or report will often be the result; with a title which may read: "A Proposed Model for the Thermal and Irradiation Stresses and Deformations in the Such-and-Such Graphite Component of the So-and-So Reactor".)

Clearly, there exists an obvious imbalance between our design analysis and materials science efforts. What is to be done? As an analyst, one can and should develop more and better communications with the materials technologists and explain more often and more clearly what is needed and what can be accomplished with the extended data. As a program director, one should try to strike a better balance between a

program's analytical efforts and its materials technology aspects. As a materials specialist, one should listen more carefully to the analyst's pleas for more applicable properties data and parameter determinations. It would appear that considerably more creativity and activity is needed in the various materials test fields. This is particularly true in the areas of devising meaningful experimental methods for obtaining suitable constitutive relations at elevated temperatures and in demanding environments. Perhaps one could suggest a general shift away from the present trends of expending the energies of our materials people in standard evaluation test data acquisition.

2. The Continuum Approach

Anyone who has viewed a piece of graphite under magnification or studied photomicrographs of the material will have the strong inclination to question the basic notion that its mechanical behavior can be represented by a continuum model. It seems to fly in the face of common sense to attempt to apply a technique that ignores the discrete mechanisms by which the material actually deforms in response to applied loads, thermal gradients, or imposed displacements. It might seem that the continuum approach can have little hope of relating the significant macroscopic parameters. Yet, that is precisely the major assumption made by (nearly) all structural analysts. At this time, it seems to be the only practical approach.

In fact, results and data necessary for the development of continuum constitutive relations must recognize and use the knowledge and details of considerations of the material's microstructure. The real questions are how to utilize the micro-data correctly, and how to apply the proper "averaging" techniques to obtain the continuum model.

It might be pointed out that the vast majority of analysts and materials scientists, and even perhaps most scientists interested in dislocations and the like, use the Fourier heat conduction law (continuum approximation) apparently almost without any question. This "law" is universally applied to graphite components and appears to be well accepted, essentially without question. Perhaps this is because we nearly always measure the parameter needed to describe this continuum model, the thermal

conductivity. Thus, based on directly determined data and considerable practical experience which indicates that an adequate model exists, we accept the continuum model and ignore the actual complex process by which heat is conducted through our graphite structures. Incidentally, it is interesting to speculate that a flaw- and void-containing material such as graphite, which apparently deforms by flaw growth and permanent volume (density) changes, might actually exhibit a coupling between the deformation and heat conduction phenomena. There are no data or investigations which bear on this point that I am aware of.

The continuum vs discrete mechanism controversy about the proper approach to inelastic material behavior is certainly not restricted to graphite. Battelle recently held a conference²⁶ in which the two schools confronted each other and the outcome was ... "Primary areas of controversy were identified, but little consensus was reached on the issues involved in treating any one aspect of inelastic behavior." Thus, it seems we must muddle along as best we can. Perhaps it is significant to note that the review of the symposium indicated a dominance by theorists. And we should also take comfort that ... "All the conferees agreed that, although a basic description of plastic flow must involve dislocation velocities and densities, the phenomenological theory of plasticity will be predominant in engineering application. The simple concept of strain will still be needed in the foreseeable future."

3. Continuum Models of Graphite Mechanical Behavior

It is true that the majority of graphite structural analyses carried out to date have used continuum mechanics. In particular, at least up to the last few years, these models were usually the linear elastic and simplified creep descriptions for the mechanical response of graphite structures. Extensions beyond these simplified approximations to complex graphite behavior have only been applied recently. Indeed, one cannot become very detailed in this area without encroaching on the speakers in Session 2 of this meeting. A brief historical sketch should suffice to set the stage, however.

As Reynolds²⁷ so aptly states... "It is important to observe that under most conditions polycrystalline graphite departs from Hooke's Law."

Characterization of the inelastic properties and nonlinear behavior of graphites has only been given serious consideration in the last decade. Most of the effort has concentrated on a room-temperature description, as an obviously logical starting point. The pioneering work of Jenkins,²⁸ an empirical approach, and Wooley,²⁹ a microstructure-based model, will be familiar to most of us. These attempted descriptions of the uniaxial, inelastic, short-time tensile and compressive behavior of graphite were a considerable improvement over linear elasticity but were a long way from a consistent thermomechanical model description of graphite mechanical response.

There have been many attempts to provide suitable correlations of various creep data for graphites. Reynolds²⁷ reviews the situation up to about 1965. The analysis of radiation-induced dimensional changes creep strain redistributions, radiation enhancement of creep rates, and related phenomena have been the bane of graphite-core power reactor designers for years. Here the need for a close interaction between experimentalist and analyst is most urgent and evident.

4. The Current Status

The current status of the work to develop practical continuum models for the behavior of graphites can at best be described as fragmented. Most of the efforts are concentrated on obtaining sufficient data and analysis for a particular graphite grade or component. The aim is usually to attempt to solve what appear to be the specific problems in a given current technology program. A notable exception is the work which has been conducted by the group at ORNL, in their Reactor Division, under the immediate direction of W. L. Greenstreet.

We will hear of some of their results during this meeting, and I will leave it to them and the other speakers in Session 2 to bring us up to date in the progress in the development of continuum mechanics models needed to perform accurate structural analyses of graphite components.

V. DIRECTIONS

If one accepts the general views outlined above, namely, that the design and analysis of optimum graphite structures for advanced technological applications are hampered by incomplete and inaccurate descriptions of the material behavior, then one is led to ask: what is to be done to bring such a body of knowledge into existence? One might even ask whether such knowledge is possible to obtain.

It would seem obvious that the magnitude of, projected large costs of, and stated urgency for several of the currently funded ambitious national programs based on graphite structures warrant more effort in the development of suitable and more accurate continuum models to describe graphite mechanical behavior. This is not to say that I believe that any specific program cannot solve a particular graphite design problem without such an effort. Certainly, clever designs, attention to the most significant parameters, conservative (non-optimum) approaches, and costly component and prototype tests can and often will achieve the desired program goals. However, we know, from the more familiar metal structures field, that the accurate account of inelastic effects (creep, plastic flow, etc.) in the analysis of structures has led to countless system performance advances and more reliable, less costly, stronger, lighter-weight structural components and provided us with structures which can operate in more rigorous environments. Admittedly, it has taken a rather long time for such knowledge to be widely applied, even in metal structure technology, but the effectiveness and necessity of application of such analyses are now widely recognized.

In graphite structural designs, the need for accurate continuum model development and application is even more compelling, both because of the brittle nature of graphite, which demands accurate stress analysis results to make accurate predictions which will avoid failures, and because of the efficiencies which will occur with more optimum, less conservative designs.

What is required for such a model development is a very closely coordinated effort between experiment and analysis; a close marriage of material scientists and applied mechanists. The most difficult aspect

of the problem is in the high-temperature experimental areas. Considerable inventiveness and creativity in the perfection of new high-temperature testing apparatus and techniques are required. It is sufficient to note that there has been no significant development effort to perfect a truly high-temperature "strain-gage" for graphite structures.

Most of us are aware that ORNL has had in its Reactor Division for a number of years, an experimentally and analytically coordinated graphite mechanics program. Some of us have even used their findings in our graphite structural analysis efforts. I would personally urge that more effort be expended for programs of this type to provide us all with a stronger base for the structural analysis of graphite components in our currently planned advanced technology applications of graphite.

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Table 1. Parameters for PSSC Power Reactor

Name	PSSC Station (Fort St. Vrain)
Owner	Public Service Company, Colorado, USA
Designer	General Dynamics Corporation
Contractor	General Atomic Division of General Dynamics Corporation
Operator	Public Service Co.
Location	Platteville, Colorado, USA
Purpose	Power generation
Type	High-temperature gas-cooled reactor
Critical Date	1972
Coolant	Helium
Inlet temp., °C	400
Outlet temp., °C	785
Pressure, atm	48
Moderator	Graphite
Control	Rods
Fuel Material	U/Th-Carbide in graphite particles coated
Geometry	Cylinders 20 ft long; 4.5-in. diameter
Enrichment	93%
Assembly	Single graphite elements
Charge	More than 2000 elements per core
Temperature, °C	
Maximum	1260
Average	815
Minimum	480
Reactor vessel	Prestressed concrete vessel
Neutron flux	Thermal
Power-Specific	
Thermal, MW	837
Net elec., MW	330
Core dens., kW/lit.	6.3
Overall station efficiency	40%
Remarks-Programs	Beginning of design, mid-1965
References	1. Nucl. Appl. 3 (1967), 601 2. BNWL-118 (Sept. 1965), DD 10 3. Nucl. Eng. (Feb. 1967), 104

Table 2. Parameters for ORNL MSBR Power Reactor

Useful heat generation, MW(t)	2,250
Gross electrical generation, MW(e)	1,035
Net electrical output of plant, MW(e)	1,000
Overall plant thermal efficiency, %	44.4
Reactor vessel i.d., ft	22.2
Vessel height at centerline, ft	20
Vessel wall thickness, in.	2
Vessel head thickness, in.	3
Vessel design pressure, psig	75
Vessel top flange opening diameter, ft	18
Core height, ft	13
Distance across flats of octagonal core, ft	14.3
Radial blanket thickness, ft	1.3
Graphite reflector thickness, ft	2.5
Number of core elements	1,412
Size of core elements, in.	4 X 4 X 156
Salt-to-graphite ratio in core, % of core volume	13
Salt-to-graphite ratio in undermoderated region, %	37
Salt in reflector volume, %	< 1
Total weight of graphite in reactor, lb	650,000
Weight of removable core assembly, lb	480,000
Maximum flow velocity in core, ft/sec	8.5
Maximum graphite damage flux (> 50 kev), n/(cm ² ·sec)	3.2 X 10 ¹⁴
Graphite temperature in maximum flux region, °F	1,284
Average core power density, w/cm ³	22.2
Maximum thermal neutron flux, n/(cm ² ·sec)	7.9 X 10 ¹⁴
Estimated graphite life, years ^a	4
Pressure drop through reactor due to flow, psi	18
Total salt volume in primary system, ft ³	1,720
Thorium inventory, kg	68,000
Fissile fuel inventory of reactor system and processing plant, kg	1,468
Breeding ratio	1.06
Yield, %/year	3.3
Doubling time, years	21

^aBased on 80% plant factor.

Fig. 1. LASL Rover Program, Nuclear Propulsion Reactor, Core of Fueled Graphite or Carbide-Graphite Composites; ~ 2000 lb of Graphite.

Fig. 2. The LASL UHTREX Reactor. Graphite Core, Helium Cooled, Fuel Temperature $\sim 1600^{\circ}\text{C}$.

Fig. 3. View of PSSC Graphite Core Reactor; ~1/2 Million Pounds of Graphite Structure.

Fig. 4. ORNL Molten Salt Breeder Power Reactor, Graphite Core Configuration.

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FATE Sacrificial Nosetip Design

Fig. 5. Typical Graphite Reentry Nose Cone Design.

Fig. 6. Typical Reentry Transient Temperature Profiles for Graphite Nose Cones.

Fig. 7. Comparison of Three Analytical Solutions for Circumferential Stresses in an Internally Pressurized Graphite Cylinder at Room Temperature: E, Elastic; P-1, Elastic-Plastic; P-2, Improved Elastic-Plastic.

MATERIAL CHARACTERIZATION

Chairman: *E. J. Seldin*, Union Carbide Corporation

CORRELATION OF PROPERTIES OF MANUFACTURED GRAPHITES*

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4802500

ABSTRACT

An homologous series of isotropic graphites was studied, and the effect of porosity on elastic moduli, thermal conductivity and electrical conductivity was evaluated. The graphites were Poco Company grades that were heated to 2900°C after sample fabrication. An approach which considered that the first 4 percent porosity was of a different type from the bulk porosity was shown to provide a reasonable explanation of the data. Measurements of modulus and strength of a second series of isotropic graphites prepared from Santa Maria flours were shown to be compatible with the results obtained on the Poco series.

INTRODUCTION

The thermal and physical properties of polycrystalline artificial graphite are functions of:

- (1) the degree of graphitization of the binder and filler phases,
- (2) the anisotropy of the bulk material and of the filler particles,
- (3) the porosity from particle packing and from accommodation voids, and
- (4) the procedures followed in fabricating and heat treating.

The evaluation of the quality of a particular manufactured graphite ideally should consist of a comparison of the measured properties with those predicted from a knowledge of these four variables. It would be particularly useful if for an experimental graphite artifact a decision could be made as to whether the variables of fabricating and heat treating were optimum for the particular binder-filler combination. The problem, of course, is that crystalline graphite is so very anisotropic in physical and thermal properties. Averaging techniques that are successful in metallic and ceramic systems face a real challenge when applied to graphites. Part of the difficulty in assessing the validity of any of these techniques is in finding graphites varying in only the property under consideration. We've had the opportunity to study one such system with

* Work done under the auspices of the United States Atomic Energy Commission.

porosity as the variable and then compare some of the results to another similar system. While the study is not complete, I'd like to show you some of the data and discuss some tentative interpretations.

RESULTS

The first system is a series of isotropic graphites obtained from the Poco company. Fifteen samples were prepared, then heated to 2900°C under flowing helium to establish a uniform degree of graphitization. Characterization of the material included measurements of density, x-ray parameters, mercury porosimetry, and optical microscopy. X-ray indications of isotropy were checked by elastic modulus, electrical resistivity and thermal conductivity measurements in orthogonal directions on several samples. Figure 1 shows the microstructures of one of the lowest and highest porosity samples. The darker areas are the epoxy mounting material. Based on a solid density of 2.25, the porosity varied from about 15 to 30 percent. A typical pore diameter was about 5 microns. Mercury porosimetry indicated that from 1 to 8 percent of the voids were inaccessible, with a very uniform pore diameter of about 0.7 micron. This number probably represents the interconnections between pores and this seems to be consistent with the observed microstructure. Table 1 lists the samples and the x-ray data. The only systematic variation seems to be in values of crystallite size, L_c , which increases from 325 Å to 440 Å with increasing porosity.

After establishing that the Poco graphites as heat treated reasonably represented an homologous series, the various properties could be correlated with porosity. The calculation of porosity from bulk density values involves a choice of the density for material of zero porosity. With metal systems the decision is simple. The steps are from polycrystalline material with voids to dense polycrystalline material to single crystals with the same density. With graphite the question is whether a filler particle is an aggregate of x-ray dense crystallites with perhaps some enclosed porosity essentially similar to particle-particle voids, or something else. Helium density, mercury porosimetry and epoxy resin impregnation experiments all indicate approximately 1 to 6% inaccessible void volume. Whether this volume is present as microscopic voids or some form of structural defect, evidence exists that indicates that this porosity is different from the interparticle porosity. For the correlations a theoretical density of 2.25 was chosen as a reasonable crystallite density, and 2.15 as the theoretical filler particle density. This approach has been suggested by various authors, and most recently discussed by Hasselman² in analyzing data obtained by Cost, et al³, on Poco graphites.

Young's and shear modulus measurements were made using thin-rod resonance techniques. Calculations of Poisson's ratios for the samples indicated that the ratio could be considered constant with porosity. Young's and shear modulus could each be fitted to an equation of the form $\frac{E}{E_0} = 1 - kP$ where the slopes were identical. The

data are shown in Figure 2. E_0 at a density of 2.15 g/cm³ where $P = 0$ is $2.5 \pm 0.05 \times 10^6$ psi, G_0 is $1.03 \pm .04 \times 10^6$ psi, $K = 1.67 \pm 0.03 \times 10^6$ psi and Poisson's ratio is 0.24. The dashed lines represent the equations $\frac{E}{E_0} = \frac{G}{G_0} = 1 - (2.3 \pm 0.1) P$, with the

values for bulk modulus calculated from the relationship $K = \frac{EG}{9G - 3E}$. Most of the correlations suggested in the literature predict too high values of modulus for a given porosity. The solid lines are the result of fitting data from porous tungsten to

Mackenzie's theory⁵ which is based on spherical pores. It is apparent that the pores in graphite are more effective in reducing the moduli than predicted by the theory.

Averaging single crystal data produces values for x-ray dense polycrystalline graphite of about $E = 8 \times 10^6$ psi, $G = 3 \times 10^6$ psi and $K = 5 \times 10^6$ psi or about three times the values at 4 percent porosity. This implies a type of porosity that is very effective in lowering the modulus. Porosity present as basal plane delaminations could produce such an effect. Figure 3 shows thermal conductivity data obtained by Paul Wagner⁶ of our group. The choice of a linear relation of thermal resistivity to fit the data produces a reasonable value of conductivity at a density of 2.15 g/cm^3 . This value of $8.9 \text{ watt/cm}^\circ\text{K}$ can be compared to a predicted value from single crystal data of about $13 \text{ watts/cm}^\circ\text{K}$ ⁷. Extrapolating the relationship to x-ray density would indicate a negative conductivity. An explanation of the decreased effectiveness of the residual 4% porosity associated with the filler particles in reducing the effective thermal conductivity is required. One argument is that the basal plane cracks postulated for the large elastic modulus reduction would be parallel to the direction of greatest heat flow and therefore much less effective in reducing the thermal conductivity.

Figure 4 shows the same data plotted as conductivity vs. porosity, Hutcheon and Price⁸ in their paper on porosity effects in the 4th Carbon Conference found a linear relation for conductivity for a series of graphites where the density was varied by re-impregnations.

Figure 5 shows the electrical conductivity data as a linear function of porosity and Figure 6 indicates that this seems to be a much better choice than a linear resistivity-porosity relationship. These data were also obtained from Wagner⁶. Extrapolating the data to X-ray dense material produces a resistivity value of $675 \mu\Omega\text{cm}$ compared to $740 \mu\Omega\text{cm}$ at 4 percent porosity. These values can be compared to an estimated $380 \mu\Omega\text{cm}$ for averaged single crystal data. This reduces to approximately the same dependence on porosity over the first 4% of porosity as indicated by the modulus data.

The second system consisted of a series of graphites prepared from Santa Maria flours. This filler produced nearly isotropic graphites with molding and extrusion fabricating methods. Samples were produced for studies of many different processing variables, using different binders and filler size distributions. The similarities with the Poco materials are (1) the nearly isotropic bulk properties, (2) the graphitizing temperature, and (3) the crystallite size range. Pore and particle size distributions were widely different with the pore size generally much larger.

A plot of the Young's modulus of these graphites is shown in Figure 7. Except for a few of the resin bonded extruded samples the line represents an upper limit of modulus at a given porosity. The line is the Poco material relationship.

Young's modulus has been shown to be related to strength measurements. Figure 8 shows compressive strength data plotted versus Young's modulus. Modulus measurements using the dynamic methods seem to be insensitive to defects that can produce low strength measurements. Assuming that this is the case, a limiting condition for strengths of this series can be indicated, such as the line shown. Figure 9 shows some flexural strength data which could have the same interpretation. An additional restraint on the line is a constant ratio of flexural to compressive strengths. The equations for the lines are $C = -5.39 + 14.7 E$ and $F = -2.31 + 6.29 E$ where the units of C and F are Ksi and the units of E are 10^6 psi. Data on thermal and electrical properties show much more scatter, but with the maximum property values approaching those of the

Poco graphites as a limiting case.

In conclusion, heat treated Poco materials seem to provide an homologous series of isotropic graphites with porosity as the only significant variable. At least one other series of isotropic graphites with similar crystallite size and heat treating temperature seems to show a reasonable correlation with this system.

There are other studies that would profit from the use of an homologous series. The temperature dependence of modulus and CTE are being evaluated and other measurements are being considered.

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Table 1. Crystalline Parameters and Porosities of Heat-Treated Poco Graphites

Specimen No.	Original Poco Grade	Bulk Density, g/cm ³	Void Fraction ^a	Crystalline Parameters		
				d ₀₀₂ , Å	L _c , Å	Anisotropy, M ^b
15	AXZ-9Q	1.539	0.316	3.365	430	0.10
14	AXZ	1.544	0.314	3.364	435	0.01
5	AXZ-QBG	1.625	0.278	3.364	420	0.17
8	AXZ-QB	1.631	0.275	3.366	375	<0.01
11	EP-1924	1.753	0.221	3.367	360	0.16
12	AXM-5Q	1.755	0.220	3.368	350	0.12
10	EP-1924	1.766	0.215	3.367	355	0.07
13	AXM-5Q	1.767	0.214	3.367	355	0.09
9	EP-1924	1.773	0.212	3.367	350	0.04
2	AXF-5Q	1.788	0.205	3.367	350	0.12
1	AXF-5Q	1.817	0.192	3.368	350	0.04
3	AXF-QBG	1.834	0.185	3.368	330	0.01
6	AXF-QB	1.896	0.157	3.368	335	0.09
4	AXF-QBG	1.900	0.156	3.368	325	0.07
7	AXF-QB	1.904	0.154	3.368	325	0.01

^aCalculated using a density of 2.25 g/cm³ for the dense material.

^bMaximum anisotropy, from $I(\Phi) = I_0 \sin^M \Phi$.

Fig. 1. Microstructures of One of the Lowest (about 15%) and the Highest (about 30%) Porosity Samples. Material was pressure impregnated with epoxy, then cathodically etched in wet hydrogen.¹

Fig. 2. Elastic Moduli of Heat Treated Poco Graphites as Functions of Porosity, Using 2.15 g/cm^3 as the Density of Solid Material. Solid lines indicate corresponding behavior of porous tungsten.⁴

Fig. 3. Thermal Resistivity of Heat Treated Poco Graphites as a Function of Porosity, Using 2.15 g/cm^3 as the Density of Solid Material. Data obtained from Wagner.⁶

Fig. 4. Thermal Conductivity of Heat Treated Poco Graphites as a Function of Porosity, Using 2.15 g/cm³ as the Density of Solid Material. Data obtained from Wagner.⁶

Fig. 5. Electrical Conductivity of Heat Treated Poco Graphites as a Function of Porosity, Using 2.15 g/cm³ as the Density of Solid Material. Data obtained from Wagner.⁶

Fig. 6. Electrical Resistivity of Heat Treated Poco Graphites as a Function of Porosity, Using 2.15 g/cm^3 as the Density of Solid Material. Data obtained from Wagner.⁶

Fig. 7. Young's Modulus of Santa Maria Filler Experimental Graphites as a Function of Porosity, Using 2.25 g/cm^3 as the Density of Solid Material.

Fig. 8. Compressive Strength of Santa Maria Filler Experimental Graphites Plotted vs Young's Modulus.

Fig. 9. Flexural Strengths of Santa Maria Filler Experimental Graphites Plotted vs Young's Modulus.

X
AIRCO SPEER GRADES FOR NUCLEAR AND AEROSPACE APPLICATIONS

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ABSTRACT

Manufactured graphite is a standard material of construction for nuclear and aerospace applications. Its increased strength with temperature coupled with refractoriness and light weight make it suitable for aerospace applications. The principal raw material for manufactured graphite is petroleum coke. Graphite can be manufactured from cokes from various type petroleum feedstock streams which not only impart particular physical, mechanical and thermal properties but which also vary its nuclear properties. Further, graphite can be manufactured with different degrees of purity permitting its use as a nuclear moderator or shield.

This paper will highlight the principal mechanical and thermal properties of manufactured graphite and discuss the range of properties obtainable commercially.

Carbon is a very plentiful element in the earth's crust and has been used in industrial type applications for over seventy years. Its principal uses through the years have been in applications such as electrodes for aluminum and steel smelting, anodes for the chemical industry, brushes, molds, crucibles and as carbon and graphite refractories. Most of these applications were satisfied by one or more of the basic properties of carbon--good electrical conductivity (Table I), thermal shock resistance, chemical inertness, high temperature stability, high or low thermal conductivity (Table II), self-lubrication, non-weldability and low cost (Table III).

As the technology of the various industries has advanced, improved carbon and graphites have been required. The newer industries such as Nuclear, Aerospace and Semi-conductor have caused major advances in the carbon industry by their demands for extreme purity--even to specific elements---extra high density, controlled strength, higher and lower coefficients of thermal expansion and improved high temperature properties. And all with smaller statistical variances than were previously available.

Today purified graphite is produced on a production scale with total ash content below 100 ppm (Table IV) in sizes up to 16 inches in diameter by 72 inches long. As one would expect less difficulty is encountered in purifying smaller cross section material and total impurity contents less than 20 ppm are common in sizes smaller than 10 inches in diameter.

The bulk density of manufactured graphite can be controlled through processing techniques. Forming pressures can be varied to regulate the basic bulk density of a graphite body and increased density is obtainable through multiple pitch impregnation which normally reduces both porosity and permeability of the graphite body.

By now it would appear that "everyone" knows that graphite strength increases with increasing temperature. Complete processing control of graphite strengths is not possible but this parameter does lend itself to statistical analysis. Principal factors affecting strength of graphite bodies are filler material type, particle size, method of forming and the bulk density. Since strength measurements are destructive less data and less reliability are available for these properties than, for example, for bulk density and specific resistance which are used for control and which can be determined non-destructively on the as formed body.

The coefficient of thermal expansion of carbon and graphite covers a rather broad spectrum (Table V). Control in a single direction appears possible over a range extending from about 1.0 to 7.0×10^{-6} per degree C. Low coefficients generally promote thermal shock resistance while the higher CTE's are required for bodies to be used as substrates for coatings to resist erosion or corrosion (Discuss Figure 1). Of course the broad range of CTE control is dependent on a choice of different filler materials and cannot be covered by any one type of filler.

We have discussed some of the general properties of graphite, now let us look at some specific data on a molded and an extruded grade.

Airco Speer Grade 8882 (Table VI) is an extremely fine grain, flaw-free, high strength, molded, electrographitic material representative of the better type materials available today. Although very popular, its use is restricted to items smaller than 24 x 20 x 9. Grade 8882 has proved satisfactory for nozzles for missile systems such as Arcas, improved Hawk, 5 inch RAP, and Sidewinder/Chapparal.

A recent review of characteristics of a variety of sizes of this grade resulted in the tabulation shown in Table VI.

Although, based on limited data, Figure 2, Thermal Expansion versus Temperature, Figure 3, Thermal Conductivity, Figure 4, Flexural Deflection Curves at Various Temperatures and Figure 5, Ultimate Strength, are found to be representative for Grade 8882. In addition to these figures we have conducted Tensile Stress-Strain, Electrical Resistivity, Tensile Creep and Tensile Shear Strength tests versus Temperature on Grade 8882. These curves are available on request.

Grade 8882 is made from standard type petroleum coke filler. An example of an extruded, near isotropic graphite, made from a high shrinkage isotropic coke is Airco Speer Grade SX-4. This grade belongs to a family of extruded, isotropic grades which has demonstrated good nuclear irradiation properties. Within this family sizes up to 18 inches in diameter have been manufactured and 14 inches in diameter by 72 inch long billets have been purified. Larger diameter billets have been purified in shorter lengths.

Grade SX-4 was developed as a substrate for chemical vapor deposited silicon carbide coatings. The average CTE of 4.1 matches quite well with that of SiC. The thermal expansion curves representative of this grade is Figure 6. Comparing these curves to those for Grade 8882, one is not too impressed that the grade is isotropic. However, compared to Figure 7 which is for a standard type petroleum coke grade, that is also extruded, one notes that the total spread of longitudinal to transverse CTE of SX-4 is only about half that for the standard coke grade.

The other normally measured properties of SX-4 appear in Table VII. The various measured properties vary with temperature in a manner we have come to recognize as typical for manufactured graphite. (See Figure 8, Thermal Conductivity, Figure 9, Flexural Deflection Curves at Various Temperatures, and Figure 10, Ultimate Strength.) Graphs of Electrical Resistivity, Tensile Stress-Strain, and Tensile Shear are available on request.

Earlier it was stated that the nuclear irradiation properties of this family of grades shows considerable potential. Figure 11 is a comparison of Transverse length change of two grades in this family compared to an NPR type graphite. The newer member, RC5, appears to have promise as an improved reactor material which can be made in the sizes normally required for this type service.

This family of extruded isotropic graphite grades also exhibits less chemical reactivity and better oxidation resistance in many industrial areas than do standard petroleum coke grades. These materials have shown potential for handling aluminum at high temperature, for fluorine gas generation and for the manufacture of nuclear fuel elements.

If you are now aware that carbon and graphite materials can be tailored to give emphasis to specific properties, this paper has been successful. Because of the long processing time for carbon and graphite and the costs associated with small batch production, carbon manufacturers will continue to draw from their stable of production materials to satisfy, as well as possible, most application requirements. For those who require special properties in carbon bodies, may I suggest you talk with the carbon industry before looking elsewhere for solutions to your high temperature problems.

Table I. Electrical Resistivities of Several Materials

Material	Electrical Resistivity (microhm-cm)
Copper	1.71 - 2.03
Steel, Cast Carbon	16
Tungsten	5.48
Molybdenum	5.17
Carbon	3500 - 4600
Graphite	500 - 1300

Table II. Thermal Conductivities of Several Materials

Material	Thermal Conductivity (cal sec ⁻¹ cm ⁻² °C ⁻¹ cm)
Copper	0.93
Steel, Cast Carbon	0.11
Tungsten	0.40
Molybdenum	0.28 - 0.35
Carbon	0.01 - 0.02
Graphite	0.20 - 0.50

Table III. Comparative Costs of Several Materials

Material	Cost (\$/in ³)
Copper	0.19
Steel, Cast Carbon	0.02
Tungsten	3.15
Molybdenum	1.37
Carbon	0.015 - 0.05
Graphite	0.024 - 0.69

Table IV. Typical Semi-Quantitative Spectrographic Analyses
For Graphite Grades

Impurity	Concentration (ppm)	
	Commercial Quality	Purified Quality
Aluminum	75	< 10
Boron	5	< 1
Calcium	200	< 10
Iron	2000	5
Magnesium	< 1	< 1
Nickel	75	< 10
Silicon	1000	30
Titanium	30	< 10
Vanadium	300	1

Table V. Coefficients of Thermal Expansion of Several Materials

Material	C.T.E $\times 10^{-6} \text{ } ^\circ\text{C}^{-1}$
Copper	18
Steel, Cast Carbon	15
Tungsten	4
Molybdenum	4.8 - 5.6
Carbon	2.3 - 7.0
Graphite	1.8 - 7.0

Table VI. Characteristics of Airco Speer Grade 8882 in Several Sizes

Sizes*	$2\frac{5}{8} \times 6\frac{3}{4}$, 4×7 , $4\frac{5}{8} \times 13$, $6 \times 5\frac{1}{2}$, $6\frac{1}{2} \times 13$, 8×13		<u>Sigma</u>
Average density, gm/cc		1.79	0.02
Flexural strength, psi	w.g. **	4725	350
	a.g.	3875	---
Tensile strength, psi	w.g.	2375	---
	a.g.	2000	---
Compressive strength, psi	w.g.	9000	---
	a.g.	9700	---
Coefficient of thermal expansion, $\times 10^{-6}^{\circ}\text{C}^{-1}$	w.g.	3.4	0.20
	a.g.	4.5	0.20
Permeability, $\times 10^{-1} \times \text{cm}^2 \times \text{sec}^{-1}$	w.g.	4.2	---
	a.g.	4.8	---

*Other sizes available. Characteristics may vary slightly from those listed.

**w.g., with grain; a.g., against grain.

Table VII. Some Properties of Airco Speer Grade SX-4 in Several Sizes

Sizes*	$3\frac{1}{2} \times 17\frac{3}{4} \times 60$, 6×72 , 14×72	
Average density, gm/cc		1.79
Flexural strength, psi	w.g. **	3500
Coefficient of thermal expansion, $\times 10^{-6}^{\circ}\text{C}^{-1}$	w.g.	4.1
	a.g.	5.2

*Other sizes available. Characteristics may vary slightly from those listed.

**w.g., with grain; a.g., against grain.

Fig. 1. Graphite Thermal Expansion; Expected Total Range with Typical Grade Range.

Fig. 2. Thermal Expansion vs Temperature, Grade 8882.

Fig. 3. Thermal Conductivity, Grade 8882.

Fig. 4. Flexural Deflection, Grade 8882.

Fig. 5. Ultimate Strength, Grade 8882.

Fig. 6. Thermal Expansion, Grade SX-4.

Fig. 7. Thermal Expansion, Grade 942S.

Fig. 8. Thermal Conductivity, Grade SX-4.

Fig. 9. Flexural Deflection, Grade SX-4.

Fig. 10. Ultimate Strength, Grade SX-4.

Fig. 11. Comparison of Transverse Length Change in Three Grades of Graphite as a Function of Fast Neutron Exposure.

THE MECHANICAL BEHAVIOR OF ARTIFICIAL GRAPHITES AS PORTRAYED BY UNIAXIAL TESTS*[†]

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Abstract—Stress-strain behaviors which are representative of nuclear-grade, or equivalent, graphites are described and discussed. Since cyclic loading test results provide important insight into the characteristics of graphite behavior, emphasis is placed on results of this type. Monotonic loading curves are also considered, and both stress versus longitudinal strain and stress versus lateral strain curves are given for monotonic and cyclic loading. The new results given in this paper are combined with results published in the literature to provide a unified description of the observed behavior.

1. INTRODUCTION

Many of the mechanical behavior characteristics of graphite have been studied. This is especially true of nuclear-grade, or equivalent, graphites as a class of materials. These graphites are generally made from petroleum-coke and coal-tar pitch, but the same overall characteristics are exhibited by graphites made from other binder and filler combinations as well. It is the purpose of this paper to bring out some of the salient features of observed mechanical behavior in a unified fashion.

Important behavioral characteristics are described in the literature. These have been identified from monotonic and cyclic loading tests, but there are certain aspects of cyclic loading behavior that either have not been studied or have not been studied in detail. Therefore, additional investigations were made to overcome this deficiency. Three types of cyclic loading were considered, including (1) cycling of specimens, which were preloaded in compression, between zero and a constant stress level less than the

preload stress value, (2) cycling in compression wherein the specimens were only partially unloaded, and (3) cycling alternately in compression and tension. Studies of the first two types have not been reported previously, and studies of the third were used to experimentally examine the validity of an often stated premise regarding reloading behavior. The results from these investigations when combined with available data provide important background information for constitutive equation development.

2. MECHANICAL BEHAVIOR

Despite material variations from grade to grade, from block to block of a given grade, and from piece to piece within a given block, there are definite characteristics of mechanical behavior associated with artificial graphite as a class of materials. Most graphites are anisotropic, and stress-strain data generally show that there is rotational symmetry of the anisotropy in an element. That is, the material may be classified as transversely isotropic.

The anisotropy is a result of the preferred orientation of the coke particles used in the manufacture and the orientations of

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the crystallites within the particles. Although the forming method, that is, molding or extruding, governs the preferred orientations of the particles, this method influences the character of the anisotropy only in terms of actual measures of deformation resistance. Therefore, the characteristics to be discussed are independent of forming method.

2.1 Synopsis of reported results*

It is common knowledge that, when a graphite specimen is loaded in simple tension or compression, nonlinear stress-strain behavior is exhibited from essentially zero stress to the failure stress. What is the characteristic of this nonlinearity? By loading to a stress level less than that at failure and unloading, one obtains a loading curve, OA , and an unloading curve, AB , as shown schematically in Fig. 1. Since the unloading

Fig. 1. Schematic drawing of a stress-strain diagram for graphite.

*Information summarized here as well as summaries of other information on graphite are given in a survey report[1].

curve does not retrace any part of the initial loading curve, the behavior cannot be classified as nonlinear elastic. The nonlinearity must be described in some other way.

It may be seen that the behavior exhibited is reminiscent of time-independent elastic-plastic behavior ascribed to metals. When the specimen is fully unloaded, there is a residual strain, OB , as for materials that undergo elastic and essentially time-independent plastic deformations. The unloading curve, AB , is also nonlinear. On loading the specimen a second time, the resulting curve is again nonlinear and a hysteresis loop is formed between the unloading and reloading curves. Loading beyond the stress corresponding to that at the unloading point, A , gives a curve which becomes asymptotic to the curve that would have existed had unloading never occurred.† There are no abrupt changes in the slopes of any of the segments of the entire curve.

The tensile strengths and the initial slopes of the stress-strain curves (elastic moduli) are generally greater in the with-grain direction than in the across-grain direction. There are pronounced differences between both stress and strain at fracture in tension and in compression. Complete stress-strain diagrams for simple tension and compression are plotted together in Fig. 2[2] to illustrate these differences in the tensile and compressive behaviors. The material is EGCR-type AGOT graphite,‡ and the data are for the with-grain (parallel to the extrusion axis) direction. Typically, fracture strains on the order of 0.1–0.2 per cent and 1.0–2.0 per cent are found in tension and compression, respectively, for nuclear-grade, or equivalent, graphites.

Arragon and Berthier[3] performed com-

†This is an often stated premise which is based on many observations. A direct experimental examination of this premise will be discussed later in this paper.

‡EGCR-type AGOT graphite is a coarse-grained, nuclear-grade, extruded graphite which was made by Carbon Products Division of Union Carbide Corp.

Fig. 2. Complete uniaxial stress-strain curves parallel to extrusion direction.

pression test studies on 216 specimens made from extruded, petroleum-coke, industrial graphite. Three types of tests were used: (1) simple compression; (2) cyclic tests (loading-unloading-reloading) in which the cycles were made from increasing stress levels spaced at equal intervals; and (3) cyclic tests between zero stress and a fixed maximum. The slopes of the stress-strain curves at the origin for continued loading (type 1) were the same order of magnitude as given by sonic measurements. Seldin[4] also found good comparisons between sonic moduli and the slopes at zero stress as measured from tensile and compressive stress-strain curves for several grades of molded graphite.

Arragon and Berthier found that cyclic compressive tests of type 2 cause the apparent density to increase. The hysteresis loops, which are formed by loading, unloading, and reloading, increase in size with increased maximum stress, and the slopes of straight lines connecting the unloading and reloading points decrease with increased stress. (The slope of a line connecting the two points of one cycle is termed the "paraelastic modulus.")

A schematic diagram, which depicts the essential features of the behavior observed from a test of type 2, is shown in Fig. 3. The envelope curve corresponds to that for simple

Fig. 3. Schematic of compressive stress-strain diagram.

compression. Arragon and Berthier[3] also discovered that straight lines drawn through the unloading point and the point of zero stress for each cycle converge at a single point as shown in the figure. The coordinates of this point are both negative (taking compression as positive), and it was reasoned that the existence of this point is a manifestation of the history of the virgin specimen.

In each case, the reloading curves asymptotically approach the envelope curve after

each cycle of loading, unloading, and re-loading. This and the other details of the behavior, as described above, are generally typical of the graphites being considered. Corroborations can be found by studying the results for extruded graphite reported by Losty and Orchard[5] and for molded graphites by Seldin[4].

In the case of test type 3, which was used in the study by Arragon and Berthier[3], each specimen was subjected to 12 cycles. During the first cycles, the total deformations at the unloading and reloading points increased with increased cycle number, but, after the sixth cycle, these deformations were essentially constant, and the hysteresis loop was retraced on each subsequent cycle. The form of the hysteresis loop remained constant

throughout the cyclic loading as did the para-elastic modulus. There was a slight increase in apparent density during cycling.

The type of behavior observed is shown in Fig. 4. These results were obtained by cycling a with-grain specimen of EGCR-type AGOT graphite in compression in the same fashion as tests of type 3 were performed by Arragon and Berthier.

The initial slopes of the stress-strain curves, or Young's moduli, for nuclear-grade, or equivalent, graphites are about the same in tension and in compression. This is generally true for both the with-grain direction and the across-grain direction. However, exceptions may be found by comparing across-grain data for some graphites. Close comparisons of tensile and compressive

Fig. 4. Cyclic stress-strain curves for a with-grain EGCR-type AGOT specimen (12 cycles).

curves for a given direction also reveal that there is a tendency toward greater deformation resistance in compression than in tension. This especially is true for the across-grain direction.

The preceding discussion was limited to stress versus longitudinal strain behavior. Stress versus lateral strain curves for these graphites have different curvatures in tension and compression; the diagrams for tension are concave toward the stress axis, while those for compression are convex. These observations were first reported by Seldin [4]. Schematic diagrams of the tensile and compressive behaviors are shown in Fig. 5.

In uniaxial compression tests, the transverse-to-longitudinal strain ratios are essentially independent of stress, yielding almost constant values. However, the ratios in tension are functions of stress, decreasing as the stress is increased. Figure 6, which shows the strain ratios for EGCR-type AGOT graphite as functions of longitudinal strain [2], provides

Fig. 5. Schematic drawings of longitudinal and lateral stress-strain curves.

Fig. 6. Strain-ratio curves from EGCR-type AGOT specimens.

a clear illustration. In this figure, the subscript 3 refers to the parallel, or with-grain direction, while the subscripts 1 and 2 refer to two orthogonal (across-grain) directions in the plane of isotropy. (Transverse isotropy is assumed.) The first of the double subscripts indicates the direction of applied stress and the second indicates the direction of induced strain. The vertical bars in the figure represent standard deviations.

When a specimen is loaded and released, the transverse residual strain is positive regardless of whether the load is a tensile or a compressive one. Thus, the volume of a specimen pulled in tension and released is increased since all linear dimensions are increased.

2.2 Additional investigations

The investigations reported in this section were made using specimens with the design shown in Fig. 7. This particular design was selected so that the specimens could be tested in either tension or compression. The 0.100-in.-dia. longitudinal hole was bored in each specimen to allow for making permeability measurements, and a smooth machine finish was used, without grinding or polishing.

Each specimen was marked for gage location using a HB grade drafting pencil to avoid scratching the surface and to provide

indications that would not be removed by heat treating the specimen at 3000°C (to be discussed later). The latter aspect is important because the specimens were heat treated and reinstrumented twice in some cases, and it was necessary to mount the gages in the same location each time. They were instrumented on opposite sides at the midpoint of the gage length with strain gages oriented in the axial and circumferential directions. Budd Metal-film, type C6-121-A, strain gages with a 0.125-in. gage length were used.

Tensile tests were performed using steel clam-shell fixtures attached to the ends and 0.20-in.-dia., stainless-steel, stranded-wire cables for transmitting the load to the specimen. The compressive tests were carried out using a subpress with a miniature load cell placed in the subpress below the specimen. By placing the load cell at this location, inaccuracies in readings due to plunger friction were eliminated.

For those cases in which the specimens were heat treated, a 100 KW, 3000 Hertz induction furnace was used for this purpose. The inside dimensions were a diameter of 9 in. and a length of 14 in., and the specimens were heated in Argon at atmospheric pressure. The time to reach the 3000°C temperature level was 45 min. The furnace was cooled from 3000 to 2500°C in approximately 15 min, from 2500–2000°C in about

Fig. 7. Drawing of test specimen.

1 hr, and from 2000°C to room temperature in 12–14 hr.

In the first series of tests, specimens from EGCR-type AGOT graphite were subjected to the following program of loading. Each specimen was loaded to a given stress level in compression and unloaded. It was then cycled ten times between zero and a stress, σ_0 , which was in the range given by

$$\frac{1}{2} \sigma_m < \sigma_0 < \sigma_m,$$

where σ_m is the maximum stress level, that is, the stress corresponding to the first unloading point. In each case, the specimen was subsequently loaded to failure. Two with-grain and two across-grain specimens were tested, and both lateral, or transverse, and longitudinal, or axial, strain versus stress diagrams were obtained. Compressive behavior was chosen because relatively large stresses and strains can be induced without failure.

Figure 8 shows typical results for both the with-grain direction and the across-grain direction. Here again it may be seen that, when the specimens were loaded to failure after being cycled, the stress-strain curves asymptotically approached extensions to the initial loading curves that would have been obtained by continued loading beyond the unloading stress, σ_m . The hysteresis loops exhibited by the with-grain stress-strain curves (Fig. 8(a)) do not change in size and coincide rather than change position along the strain axis during the first few cycles, as was shown in Fig. 4. Except for very slight deviations in the initial portions of the first few reloading curves, the hysteresis loops are again retraced on each cycle for the across-grain stress-strain curves (Fig. 8(b)).

Partial unloading characteristics were examined using a second set of four with-grain and four across-grain EGCR-type AGOT graphite specimens. A specimen was loaded in compression to a given stress level, σ_m , and unloaded. It was again loaded to a higher compressive stress level, σ_n , unloaded,

reloaded to σ_n a second time, and unloaded. A companion specimen was then loaded in compression to σ_m , partially unloaded, reloaded to the higher compressive stress level, σ_n , partially unloaded, reloaded to σ_n , and unloaded.

The stress-strain diagrams for two companion across-grain specimens are shown in Fig. 9. These are illustrative of the results obtained.

The cyclic behaviors of the fully unloaded specimens follow the patterns described earlier. An examination of the stress versus longitudinal strain curves reveals several features. The hysteresis loops increase in size and the paraelastic moduli decrease with increase in unloading stress level, the total deformations increase upon loading a second time to the higher compressive stress level, σ_n , and the reloading curves become asymptotic to extensions of the initial loading curves when the specimens are loaded beyond σ_m . The stress versus lateral strain curves are very similar in character to the stress versus longitudinal strain curves.

In the case of partial unloading, hysteresis loops are again formed, and the same general features that were observed for full unloading may be seen. The hysteresis loops are smaller, but the segments of each curve are entirely nonlinear. The asymptotic approach to the extension of the initial loading curve in each case corresponds to that for full unloading.

Since the details of the asymptotic approach feature are important to the development of mathematical analogs to the material behavior, the curves obtained from partial unloading test series were carefully studied to provide quantitative data. When a specimen is loaded to a stress level such as σ_m , unloaded either fully or partially, and reloaded, the reloading curve undergoes an accelerated change in slope as the stress approaches the stress, σ_m , at the point of unloading. This accelerated change persists until the reloading curve essentially begins

Fig. 8. Cyclic curves for two preloaded EGCR-type AGOT specimens.

to trace what would have been the extension to the initial loading curve provided unloading had not occurred. The quantitative data obtained were the stress values at the extremities of the accelerated change regions. The lower limit is denoted by σ' and the

upper limit is referred to as σ'' .

The values for σ' and σ'' were obtained by plotting the ratio of longitudinal strain to stress as a function of stress. The data on either side of σ_m are described by straight lines with greatly different slopes. The

(a) Full Unloading

(b) Partial Unloading

Fig. 9. Cyclic curves for fully and partially unloaded across-grain EGCR-type AGOT specimens.

transition region then corresponds to the region where accelerated change in slope of the stress-strain diagram occurs, and the limits may be determined. A typical example of the plots used for determining σ' and σ'' is shown in Fig. 10.

The two stress levels, denoted by σ' and σ'' , and the value of σ_m for each stress versus longitudinal strain curve are listed in Table 1. Also included are the values for the ratios σ'/σ_m and σ''/σ_m .

The dots on the stress-strain diagrams of

Fig. 9 mark the limits of the transition regions. It may be seen from the table that there is consistency between the values. The value of σ_m is the same for all cases. In five of the eight cases, the σ'/σ_m ratios are the same, and all ratios fall within the range from 0.93 to 0.97. The upper extremities of the segments for full unloading are at higher stress levels than those for partial unloading, which shows that asymptotic approach is achieved sooner in the latter case. For full unloading, the σ''/σ_m ratio ranged from 1.04

Fig. 10. Plot for determination of the extremities of the accelerated change in slope region for a fully unloaded across-grain EGCR-type AGOT specimen.

Table 1. Data obtained from curves for partial unloading studies

Specimen number	Orientation	σ_m (psi)	σ' (psi)	σ'' (psi)	σ'/σ_m	σ''/σ_m
101	WG	2000	1850	2130	0.93	1.07
101a	WG	2000	1950	2040	0.97	1.02
102	WG	2000	1900	2130	0.95	1.07
102a	WG	2000	1900	2050	0.95	1.03
103	AG	2000	1850	2080	0.93	1.04
103a	AG	2000	1900	2020	0.95	1.01
104	AG	2000	1900	2100	0.95	1.05
104a	AG	2000	1900	2025	0.95	1.01

to 1.07, while this ratio ranged from 1.01 to 1.03 for partial unloading.

The final test series reported herein was used in an attempt to experimentally verify the asymptotic approach premise and to provide data on cyclic behavior under alternate compressive and tensile loadings. Studies on the validity of the asymptotic

approach premise were motivated by Seldin's [4] demonstrations that test specimens can be made to reproduce their original stress-strain responses in the longitudinal and lateral directions by heat treating them at their graphitization temperatures. Provided it is generally true, this is a revolutionary discovery in graphite testing, especially in

the potential it provides for removing uncertainties in data analyses and interpretations that arise because of the almost inevitable variations in graphite properties.

Pursuant to making the final studies regarding the cyclic behavior of graphite, Seldin's method for removing deformation history was examined using specimens made from ATJ, RVD, and EGCR-type AGOT graphites.* The maximum particle sizes are 0.006, 0.015, and 0.032 in., respectively. The first two are molded graphites. Each specimen was subjected to the following sequence of steps.

1. Heat treat at 3000°C for 60 min.
2. Load in tension to a predetermined stress level and unload.
3. Heat treat at 3000°C for 60 min.
4. Load in compression to a stress level greater than in Step 2 and unload.
5. Heat treat at 3000°C for 60 min.
6. Load in tension to the stress level of Step 2 and unload.

The first heat treatment was used to establish the graphitization temperature in each case.

Stress-strain curves for one with-grain and one across-grain specimen of EGCR-type AGOT graphite are plotted in Fig. 11, where both the longitudinal and lateral strains are shown. All stresses, tensile and compressive, and the corresponding longitudinal strains are plotted as positive; the transverse data are plotted in an analogous manner. Note that the two strain scales are not the same; hence, the differences between lateral strain curves are accentuated. Curve No. 1 is for the first loading in tension, No. 2 is for the compressive loading, and No. 3 is for the second loading in tension.

The success of the heat treatment method for removing deformation history is determined by comparing curves 1 and 3 for each

specimen. The curves for the with-grain specimen show that the heat treatment was reasonably effective. It was less effective for the across-grain specimen, however.

The heat treatment method was more effective in restoring the original stress-strain behavior of ATJ and RVD graphite specimens than it was in the case of EGCR-type AGOT graphite. Agreement between curves for the first and second tensile loading was very good for both with-grain and across-grain specimens of ATJ graphite and for with-grain RVD specimens. The corresponding agreement was not as good for across-grain RVD specimens, but it was better than that shown in Fig. 11(b) for the across-grain EGCR-type AGOT specimen.

The results for the three graphites showed that, in general, heat treating does almost entirely remove the prior deformation history so far as stress-strain response is concerned. Although there were small decreases in deformation resistance between the first and second tensile loadings, the agreement between the curves was generally very good. The greater differences between the first and second tensile loadings for across-grain RVD and AGOT specimens may have been associated with opening of microcracks too large to be healed by heat treatment. This conjecture is the most plausible for the coarse-grained AGOT material.

Some of the behavioral aspects which were discussed in the previous section may be seen from the curves of Fig. 11. The deformation resistances are essentially the same in tension and in compression, with a very slight tendency toward greater deformation resistances in compression than in tension for the across-grain direction. The curvatures of the stress versus lateral strain curves in tension are different from those in compression but the initial slopes are nearly equal. Finally, positive residual lateral strains are incurred as a result of loading in tension.

In addition to the investigations just

*ATJ and RVD graphites are manufactured by Carbon Products Division of Union Carbide Corp.

(a) With-Grain

(b) Across-Grain

Fig. 11. Stress-strain curves for two EGCR-type AGOT specimens.

described, studies were made to examine the influence of hold time at 3000°C. EGCR-type AGOT graphite specimens were used, and the hold times were 20, 30, and 40 min (in addition to the 60 min hold time used in obtaining the results already discussed).

The results were inconclusive because no definite trend with hold time was established. However, it was decided that the longer hold time is preferred to assure greater possibility for reproducible results.

Returning to the test series for examining

the asymptotic approach feature and for obtaining data on cyclic behavior under alternate compressive and tensile loadings, three (two with-grain and one across-grain) EGCR-type AGOT specimens were tested. After an initial heat treatment at 3000°C for 60 min, the first loading for each specimen was used to establish one-cycle envelope curves. Each was loaded in compression near that required for failure, unloaded in tension to a given stress level, again near that required for failure, and unloaded. The envelope curves for one of the with-grain specimens are shown as short-dashed lines in Fig. 12.

During preliminary tests it was found that a specimen will fail in tension when the tensile strain, as measured from the zero stress point, corresponds to that at failure for a virgin specimen loaded in tension only, regardless of the position of the zero stress point. Thus, considerable care must be used in selecting maximum tensile stress levels to avoid fracture in tests of this type.

Each specimen was heat treated a second time under the same conditions as above and retested. It was loaded in compression to a

given stress level, unloaded, loaded in tension, unloaded, loaded in compression to a higher stress level than the first, and unloaded. The corresponding curves for the with-grain specimen are shown by the long-dashed lines in Fig. 12. The apparent discontinuities in the slopes of the curves between the tensile and compressive portions were caused by changing fixtures to apply loads of different signs.

Finally, the specimens were heat treated again and the loading sequence was repeated using lower compressive stress levels. The solid curves in the figure were obtained in this way. The maximum tensile stress level was essentially constant for all loadings on a given specimen.

The results for the other two specimens were similar to those shown in Fig. 12. In all cases, the stress level for reloading approached that at the unloading point before a transition in slope occurred. The curvature of a stress versus longitudinal strain curve generally changed rapidly in the immediate vicinity of the unloading point, while the change for a stress versus lateral

Fig. 12. Multi-cycle curves for a heat-treated EGCR-type AGOT specimen (with-grain).

strain curve occurred over a much wider stress interval. Behavioral details for the stress versus longitudinal strain curves that are typified by the solid lines in Fig. 12 were difficult to detect in all cases because of the small scale. In fact, the initial loading and reloading curves were separated in Fig. 12 for clarity. The discontinuities in slope attributable to changing of loading fixtures were apparent in almost all cases. The hysteresis loops and other aspects of the behavior were in accord with the results from other cyclic tests.

Consider the stress versus longitudinal strain curves. The asymptotic approach feature at the lower stress levels was observed, but there were small deviations as the unloading stress level was increased. For the three specimens tested, the upper portions of the dashed curves were slightly above the envelope curve in two cases and a little below this curve in one case, that is, the one for which the results are shown in Fig. 12. On the whole, asymptotic approach to an initial loading curve was demonstrated by these data.

Additional support is given by the stress versus lateral strain curves for the with-grain specimens that are shown in Fig. 12. The agreement of subsequent loading curves with the envelope curve is excellent. The remaining two specimens gave subsequent loading curves which fell below the envelope curve in each case. However, these subsequent loading curves appeared to define envelope curves although they were different from those established during the first loadings. Hence, the results from these two specimens indicate that asymptotic approach is at most closely approximated. Conversely, because the restoration of the original stress-strain response by heat treating was shown to be less than perfect by the studies made to examine this technique, fully satisfactory agreement would be difficult to achieve.

A better selection for studies of this kind would have been ATJ graphite specimens since heat treatment was shown to be more

effective for this than the other two materials examined. As demonstrated here, discovery of the heat treatment method allows for detailed comparisons where none could be made formerly.

3. CONCLUSIONS

This paper describes the stress-strain responses of nuclear-grade, or equivalent, graphites under monotonic and cyclic loading conditions. Both stress versus longitudinal and stress versus lateral strain data are considered, and it is shown that consistent patterns of behavior are identifiable from the results. These include differences in deformation resistance in tension and compression and differences in strain-ratio data for the two types of loading.

When a specimen that has been preloaded in compression is cycled in compression between zero and given maximum stress levels, three behavioral patterns are observed. For the case in which the maximum stress levels are continually increased and all are greater than the preload value on each subsequent cycle, the hysteresis loops become larger and the paraelastic moduli decrease with increasing maximum stress. Cycling between zero and the preload stress level produces hysteresis loops that do not change with cycle number and the paraelastic moduli remain the same. However, the loops translate along the strain axis for the first few cycles, after which the loops coincide. Finally, cycling between zero and a fixed maximum value which is less than the preload stress produces hysteresis loops that are essentially invariant both in size and location with cycle number. In the case of a specimen which is loaded, partially unloaded, and reloaded, hysteresis loops are formed by the stress versus longitudinal strain and stress versus lateral strain curves. Also, the reloading curves approach what would have been extensions to the initial loading curves more rapidly than for the case of full unloading. The approach to the extension of a

continued loading curve on reloading is often assumed. Here, this postulate is shown to be supported by data obtained from tests designed to examine its validity.

The use of heat treatment at the graphitization temperature essentially removes the effects of prior deformation, so far as stress-strain response is concerned. However, these heat treatments were more successful in removing the prior deformation histories of ATJ and RVD graphites than of EGCR-type AGOT graphite. Careful use of this technique is shown to be of unique benefit for making comparisons of the stress-strain behaviors of a single graphite specimen under different loading histories.

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MECHANICAL AND THERMAL PROPERTIES
OF GLASS-LIKE CARBONS

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ABSTRACT

The term "glass-like carbons" is a generic term describing a family of amorphous isotropic carbon materials which have very high mechanical strength, very low permeability, and very good chemical and erosion resistance. The high mechanical strength is related to structural defects such as intercrystalline bonding and the lack of crystalline order. The superior chemical properties are a function of low permeability and low surface area.

The properties of these materials can be varied not only by heat-treatment or stress graphitization, but also by variation of the precursor material or by the use of additives. Thus, if one wishes to use a material of this sort for a specific application, one can consider the design requirements and vary the properties accordingly.

For example, a variation in precursor formulation has been used to enhance the mechanical properties of these materials as produced in our laboratory. Specifically, the tensile strength has been increased from 17,000 to 29,000 psi and the compressive strength from 128,000 to 200,000 psi.

For extremely severe hyperthermal environments, it is desirable to increase the thermal conductivity of the material while maintaining mechanical properties equivalent to that of high strength graphite. By using fine graphite powder in a glassy carbon matrix, it is possible to promote graphitization and nucleate crystalline growth and rearrangement. The resultant product is a fine grain ($5\ \mu$) isotropic graphite in a glassy carbon matrix. The product has a thermal conductivity approaching that of graphite ($0.112\ \text{cal/cm}^2\cdot^\circ\text{C}\cdot\text{sec}$) with mechanical properties (tensile strength = 8,600 psi and compressive strength = 26,600 psi) as good as that of conventional high strength graphites. The high-temperature erosion characteristics are superior to those of conventional graphites, and the thermal shock characteristics are greatly enhanced.

Thus, it is possible to alter the characteristics of this class of materials to meet specific design requirements.

INTRODUCTION

Glass-like carbons, unlike polycrystalline graphites, are an isotropic form of carbon which have unusually high strengths which apparently are attributable to their disordered structure, extremely fine crystal structure and pore structure. As indicated by Fischbach,¹ this strength appears to be related to rigid intercrystalline bonding and strong intercrystalline constraints.

LMSC GLASS-LIKE CARBON DEVELOPMENTS

Standard and Advanced Grades

By variation of the polymer precursor to increase intercrystalline bonding, we have been able to effect a substantial increase in the mechanical properties of these materials as summarized in Table 1. As shown by this table, the mechanical properties of all grades of these materials are high, but substantial improvements can be made by changes in formulation. The tensile strength is as high as 29,000 psi and drops to only 26,000 psi after heat-treatment to 3000°C. The maximum flexural strength is 22,900 psi and the maximum compressive strength is 200,000 psi and drops to 150,000 psi in the 3000° heat-treatment conditions. The impact strength, Young's modulus and shear modulus are not substantially affected by heat-treatment. The material is hard but becomes slightly softer with high-temperature heat treatment.

These unusual mechanical properties are believed to be associated with the detectible variation in microstructure, specifically the pore size, density, and degree of graphitization. Variations of the physical parameters as a function of heat-treatment condition are given in Table 2. As we all know, glass-like carbons are formed by the thermal degradation of thermosetting resins. In our material, maximum molecular rearrangement occurs at 500°C, as opposed to 700°C reported by Fitzer³ for mixtures of phenolic and furfuryl alcohol. At this point, density is at its lowest, 1.25 gm/cc, and pore size is at its largest, 500 to 700 Å. Upon further heat-treatment, shrinkage occurs; there is a decrease in density, the pores become very small, and the material becomes impermeable. At 1000°C, the density is at a maximum, 1.52 gm/cc, and the pore size is at a minimum. Further heat treatment causes some decrease in density and a slight increase in the pore size. Heat treatment to 2000 and 3000°C causes some graphitization as shown by the decrease in interlayer spacing and increase in apparent crystallite size.

The porosity observed at 500°C is shown in an electron micrograph of a surface replica in Fig. 1. These small depressions, which we believe represent an open channel porosity, were the only evidence of any type of pore observed in the partially degraded plastic. No evidence of any structure was found by scanning electron microscope.

The structure of the glassy carbon is illustrated by transmission electron micrographs (Fig. 2). At 1000°C heat-treatment condition, the structure may be described as a random network of carbon atoms. The fine porosity calculated from small angle x-ray diffraction measurements is substantiated by the electron micrographs. Upon heat-treatment to 2000°C, the structure is much more clearly delineated and there is better definition between void areas and regions of high carbon density. The appearance of the material is compatible with the model suggested by the x-ray data.

Comparing the curves for density and tensile strength as a function of heat-treatment condition (Fig. 3), we see that the two curves coincide in shape below 1000°C. Thus, under these conditions, the tensile strength must be directly related to density and pore structure. However, above 1000°C the density decreases and the

pore size increases, yet the tensile strength increases to a maximum at a 2000°C heat-treatment condition and decreases slightly at 3000°C.

Since the high tensile strength of glass-like carbons is believed to be associated with structure defects in the form of cross linking and intercrystalline bonding which inhibit the high basal plane shear compliance, S_{44} , normally found in graphitic materials, such defects must be at a maximum in the 2000 grade material. The growth of crystallite size and increased order becomes significant only above 2000°C, so that the deterioration of tensile strength to values more nearly like that of graphite become significant only at the higher heat-treatment temperature.

The thermophysical properties of the two standard grades of glassy carbon are summarized in Table 3. As previously indicated, the porosity consists of fine closed pores and the material is quite impermeable. The electrical resistivity is an order of magnitude lower than that of graphite. The specific heat is about the same. The thermal conductivity is very low. The thermal expansion coefficient and the spectral emissivity are similar to values found in graphites.

Graphite-Filled Glass-Like Carbon

Because of the very low thermal conductivity, the question of thermal shock arises. In most applications, thermal shock has not been a problem. For example, small nose tips have been tested at very high temperatures and enthalpies without thermal shock. However, at very high stagnation pressures such as 70 atm, samples may fail in shock.

We set out to modify the characteristics of our material to make them more graphitic. Our approach was to use additions of fine graphite filler. Our goal was to increase the thermal conductivity while maintaining strength properties equal to that of graphite and maintaining superior ablation characteristics.

The effect of heat treatment on thermal conductivity is shown in Fig. 4. The thermal conductivity increases with increasing filler addition and increasing heat-treatment time. However, for each percent addition, the effect of heat treatment decreases with time.

The microstructure as a function of heat-treatment condition is shown in Fig. 5 and indicates that the matrix around the particles becomes more graphitic with time and that the polycrystalline phase increases with time at temperature.

X-ray diffraction data and calculation of apparent volume fraction of crystalline material indicate that during heat treatments the effective volume fraction of graphitic material increases in a manner such that the process is controlled by the rate of volume diffusion. Such additions apparently promote graphitization and nucleate crystalline growth and rearrangement.

The mechanical properties of the filled material decrease with heat-treatment time. For example, the flexural strength approaches a limiting value of about 6000 psi and decreases with increasing filler addition (Fig. 6). Polycrystalline graphite tested in the same way had a flexural strength of 6600 psi. Similar effects were obtained for the tensile strength of these materials (Fig. 7). After 12 hours of heat-treatment, the tensile strength approached that of polycrystalline graphite tested with the same test configuration. Based on these studies, it was decided that a 2-hour cycle was an optimum heat-treatment condition to maintain the desired mechanical properties.

The properties of the standard glass-like carbon versus the graphite-modified material are listed comparatively in Table 4. The density of the modified material is slightly higher and is directly proportional to the weight fraction of additive. The mechanical properties are considerably lower, but nevertheless compare favorably with polycrystalline graphite. The thermal conductivity of the material has been

increased by a factor of 10. The ratio of strength-to-modulus has been slightly improved as has the strain-to-failure. It is therefore estimated that the thermal shock resistance has been increased by a factor greater than 10.

SUMMARY

The properties of glass-like carbon can be varied to meet specific design requirements, either by varying the precursor materials or by the use of additives. Formulations which promote structure defects such as cross-linking and intercrystalline bonding result in an increase in mechanical strength. Utilizing this approach, the tensile strength has been increased from 17,000 to 29,000 psi and the compressive strength from 128,000 to 200,000 psi. The use of heat-treatment conditions which promote increasing crystalline order results in a slight decrease in strength.

The use of graphite filler additions promote crystalline growth and rearrangement. The resultant product is a fine grain isotropic graphite in a glassy carbon matrix. The use of such additions result in a decrease in tensile strength but an improvement in the strain-to-failure. The thermal conductivity is greatly increased, resulting in improved thermal shock characteristics.

Thus, it is possible to alter the characteristics of this class of materials to meet specific design requirements.

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Table 1. Mechanical Properties of Glass-Like Carbons

Property	<u>LMSC Glass-Like Carbons</u>			
	<u>2000 Grade</u>		<u>3000 Grade</u>	
	Standard	Advanced	Standard	Advanced
Tensile strength (psi)	17, 000	29, 000	15, 000	26, 000
Flexural strength (psi)	18, 100	22, 900	13, 100	20, 000
Compressive strength (psi)	128, 000	200, 000	86, 600	150, 000
Impact strength (in.-lb/in. notch)	1.2	—	1.2	—
Poisson's ratio	0.12	—	0.10 est	—
Young's modulus (10^6 psi)	4.0	—	3.4	—
Shear modulus (10^6 psi)	1.65	—	1.4 est	—
Hardness, Vickers	200	—	90—100	—

Table 2. Physical Parameters as a Function of Heat-Treatment Condition

<u>Heat Treatment Temperature</u> (°C)	<u>Density</u> (gm/cc)	<u>Pore Size</u> (Å)	<u>X-Ray Diffraction Characteristics</u>	
			<u>Interlayer Spacing</u> (Å)	<u>Crystallite Size</u> (L_c , Å)
500	1.25	500—700 ^a	—	—
750	1.38	<100 Å ^a	—	—
1000	1.52	22—30 ^b	3.60—3.65	10
2000	1.50	36—40 ^b	3.56	20
3000	1.42	50 ^b	3.44	100

^aAs determined from electron microscopy.

^bAs calculated from small angle x-ray scattering measurements by W. S. Rothwell.²

Table 3. Thermophysical Properties of Standard LMSC Glass-Like Carbons

Property	Material Grade	
	2000	3000
Gas permeability (cm ² /sec)	10 ⁻¹⁰ - 10 ⁻¹² est.	10 ⁻⁷ - 10 ⁻⁹ est.
Electrical resistivity (10 ⁻⁴ ohm-cm)	60	40
Specific heat (cal/g/°C)	0.18 at 38°C 0.48 at 927°C	Unchanged
Thermal conductivity (cal/cm·°C·sec)	0.010	0.012
Thermal expansion coefficient (10 ⁻⁶ /°C)	4.2	5.0
Spectral emissivity at 0.65 μ	0.80	0.76

Table 4. Properties of Graphite-Filled Versus Unfilled Glassy Carbon

Property	Standard 2000 Grade	Graphite Modified Glass-Like Carbon
Heat treatment temperature (°C)	2,000	2,500
Density (gm/cc)	1.45	1.56
Tensile strength (psi), S	17,000	8,600
Flexural strength (psi)	18,100	8,600
Compressive strength (psi)	150,000	26,600
Young's modulus (psi), E	4.0	1.4
Poisson's ratio, μ	0.12	0.10
Thermal conductivity, K (cal/cm ² ·°C·sec)	0.010	0.112
Thermal expansion, α (10 ⁻⁶ /°C)	3.4	3.0 est.
S/E	4.25	6.15
Strain-to-failure (%)	0.12	0.33

Fig. 1. Electron Micrograph of Replica of 500 Grade Precursor.

1000-8

2000-2

Fig. 2. Transmission Micrograph of Two Grades of LMSC Glass-Like Carbon.

Fig. 3. Density and Tensile Strength as a Function of Heat Treatment Temperature.

Fig. 4. Effect of Heat Treatment on Thermal Conductivity of Graphite Filled Glassy Carbon.

Fig. 5. Effect of Heat Treatment on Microstructure of Graphite Filled Glassy Carbon.

Fig. 6. Effect of Heat Treatment on Flexural Strength of Graphite Filled Glassy Carbon.

Fig. 7. Effect of Heat Treatment on Tensile Strength of Graphite Filled Glassy Carbon.

X DYNAMIC BIAXIAL AND ELEVATED-TEMPERATURE PROPERTIES
OF ATJ-S GRAPHITE

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ABSTRACT

This paper describes the techniques of and results from tests on ATJ-S graphite in which the fracture surface in bi-axial stress and the fracture stress at various temperatures and heating rates were determined. The technique developed to load tubular specimens to fracture in four stress-ratio quadrants involves applying an axial load and a circumferential pressure simultaneously at controlled strain rates up to approximately 10/sec. To study the high-heating-rate high-temperature properties, tensile specimens are heated by direct current at various rates up to 1000°F/sec and then pulled to fracture at strain rates up to about 10/sec.

Results from the biaxial experiments show that graphite follows a Mohr (internal friction theory) fracture criterion, where the critical shear stress at yield (or fracture in this case) is a function of the normal compressive stresses acting on the slip planes. Premature buckling caused by difficulties with the experimental technique in the compression-compression quadrant prevented obtaining valid data. Material scatter overshadows any strain rate sensitivity of combined-loading fracture stresses for strain rates from 10^{-3} to 10/sec. Results show that this material is unable to withstand any principal tensile strains in excess of about 1 to 1.5% strain, regardless of the loading state.

Results from the high-heating-rate tests showed that no significant heating-rate or time-at-temperature effects on fracture stress were encountered up to near 5500°F. Above this temperature, time effects appear important. Surface effects at temperatures near 6000°F caused localized porosity or pitting, thus reducing the true cross section of the test specimen. Interpretation of stress under these conditions from axial load measurement was difficult.

This paper summarizes the results of several years of investigation on the dynamic mechanical properties of ATJ-S graphite, and perhaps it will answer some of the questions that were raised in Session 1 of this conference. Four techniques will be reviewed, and test results from each will be presented.

Schematics of the devices used for the first two techniques are shown in Fig. 1, and photographs of these two devices are shown in Fig. 2. Although they were not developed specifically to test graphite, they are reviewed briefly. The first is a medium strain rate machine. This is a quasi-static device, capable of loading materials in uniaxial tension or compression, at strain rates from 10^{-3} /sec to $\sim 10^2$ /sec, the maximum rate being that which would generate stress waves into the machine. A double-ended piston is driven with air through an orifice to control the strain rate, and the test is started by actuating a fast-acting valve. Load is measured with a load bar, mounted directly above the specimen. The strain is measured normally using optical extensometers or strain gages mounted directly on the specimen. The medium strain rate machine shown in Fig. 1 is capable of performing elevated-temperature tests up to $\sim 1000^\circ\text{F}$, with heatup times of the order of 15 to 20 min., or longer. Specimens are heated by radiation from the quartz lamps.

The second device is a Hopkinson bar. Briefly, it uses one dimensional elastic wave analysis to determine the stress and particle velocity on the specimen faces as a function of time. The driver bar impacts the weighbar, and sends an elastic wave down this bar through the specimen and into the anvil bar. Measurements are taken from strain gages on the two bars, and from those measurements stress and particle velocity at each face, and subsequently strain, can be determined as a function of time. The maximum strain rate on this device depends on the impact velocity, the specimen length, and the assumption of uniform one-dimensional stress in the specimen. Nominally, this is of the order of 10^3 /sec.

Figure 3 is a composite stress-strain curve for ATJ-S graphite from tests taken on these two machines. Note the low strain rate sensitivity and directional effects and the absence of any indication of temperature effects. ATJ-S exhibits relatively low anisotropy and strain rate effects, at least up to rates of 10^3 /sec as shown here. On this graph, tension is plotted in the same direction as compression, the modulus being very close to the same for compression and tension. Usually, of course, they are plotted in opposite directions. Results up to 700°F were essentially identical (within the material scatter) to room-temperature results.

The next two techniques were developed on an Air Force contract entitled MARS (Material Response Studies). Figure 4 is a schematic of a device to test materials in biaxial stress at strain rates from 10^{-3} /sec to $\sim 10^2$ /sec. Tubular specimens (about 2.5 in. O.D., 6 in. long, with a 1/4-in. wall) can be tested in all four stress quadrants. A central piston (in the 12.5-in.-dia. chamber) operating on the same principle as the piston in the medium strain rate machine described above, provides axial load, either compression or tension; two auxiliary pistons provide the driving force to oil for circumferential loadings. Proportional strain or proportional stress tests can be conducted.

The device does not have any elevated-temperature capability, but a hydrostatic pressure bomb can be added to the system to examine the effect of pressure on the yield surface of materials. Figure 5 is a photograph of this machine, which is quite large.

Figure 6 is a plot of the biaxial fracture stress surface for ATJ-S graphite at room temperature. Note the lack of strain rate sensitivity, similar to the uniaxial data. The data points labeled B are believed to be conservative, due to premature buckling in the compression-compression tests. This is a severe problem, and it tends to be more severe closer to the tangential axis. The data points in the tension-tension quadrant are believed to be conservative also, due to end effects caused by gripping action on the specimen.

From Fig. 6, the behavior of the material appears similar to that of other brittle-type materials. Uniaxial compression and tension results from materials taken from the same billet as the tubes were used in two analytical expressions — the Mohr and the Norris criteria, as shown — for the failure surface. These expressions are modifications of the well known Tresca and Mises criteria for failure in orthotropic materials. Within the data points shown, agreement is good for either one.

The characteristics of this surface will change with temperature, as will be shown later by Jortner and Babel (in the session on "Fracture") on another graphite material, and with mean normal pressure. Large effects of hydrostatic pressure on failure in rocks are well known. The effect on graphite has not been characterized, to my best knowledge. Recently, we conducted a series of triaxial tests up to 60,000 psi confining pressure, and interestingly no failure was observed up to ~40,000 psi compressive stress. When this is compared with the ~12,000 psi pressure stress here, it is quite obvious that hydrostatic pressure effects on this material — and I would assume materials like it — are very serious. In addition, although strains well above 20% were induced in the specimens during these tests, residual strains of only 4 to 5% were recorded after the tests, with no observable cracking; there were no microcracks observable on the surface.

The strains associated with Fig. 6 are plotted in Fig. 7, the fracture strain surface for ATJ-S graphite, which shows that this material cannot withstand any principal tensile strain greater than about 1 to 1.5%, regardless of the loading state.

The device used for the last technique is shown schematically in Fig. 8. Here elevated-temperature tests (up to ~6500°F) have been conducted in uniaxial tension or compression at strain rates up to ~10/sec. In addition, short-time-at-temperature (or high-heating-rate) effects have been studied. Direct current is used to heat the specimens to temperature and maintain them at temperature for any length of time. An environmental chamber surrounds the specimen grip area to allow evacuating any air or back-filling with inert gas, such as helium or argon. A graphite reflector is used to minimize radiation heat losses on the specimen. This becomes a very severe problem above roughly 5000°F when heating by direct resistance. Loading is provided in the same way as in the medium strain rate machine described above. In addition, this machine has been modified to allow a rapid reverse loading, to study Bauschinger effects and volumetric changes at temperature.

In this device also, load is measured with strain gages mounted on the removable bar above the specimen. Precise pretest alignment can be accomplished with an adjusting system to minimize bending stresses in the specimen during loading. Strain can be measured using a laser extensometer technique that was developed, in which an argon laser is used to scan the gage section; the beam is reflected from targets to a photomultiplier system to determine strain-time history.

Tantalum and hafnium deposited onto the gage section of the graphite specimens using rf sputtering proved excellent for gage marks. The argon laser is used in order to provide a scanning source outside the spectrum of the hot samples. Figure 9 is a photograph of the system, showing the power supply, the testing device and controls, and the recording system.

Figure 10 shows the results of tests conducted to determine the effects of heating rate on the fracture strength of ATJ-S. All tests were conducted at a strain rate of $\sim 10/\text{sec}$ (loading times were tens of milliseconds), with emphasis in this study placed on temperatures above 5000°F . Heating rates were varied from approximately 10 to $1000^\circ\text{F}/\text{sec}$., at different temperatures and different hold times at temperatures. The error bands indicated were due primarily to the scatter in fracture stress that was observed on the material and to the uncertainty in temperature measurement, which in turn was primarily due to the drastic changes in surface characteristics with time, which affect the true emissivity. This was quite a problem, especially at the higher temperatures.

When compared with previously published long-time-at-temperature data (dashed line), the data in Fig. 10 shows a heating rate effect above about 5500°F , with certain unexplainable effects below this. As an example, at 6000° a significant strength, of the order to 5000 psi or above, was observed, where long-time data essentially shows no strength at all. One possible explanation for this is that extremely time-dependent localized heating takes place at these high temperatures, both at the surface and internally in the specimen. In Fig. 11, note the larger voids in the heated sample in the upper photomicrograph, in comparison with the smaller voids in the raw material below. This was quite significant in several of the samples we checked. Additional surface phenomena were observed while viewing the surface through the optical pyrometer used to measure temperature, and one could actually see localized pitting taking place as a function of time. Thus obviously the cross-sectional area is an unknown in the determination of stress.

In conclusion four experimental techniques have been reviewed, two of which are unique for their short-time, high-strain-rate characteristics. Test results on ATJ-S graphite show it to behave as a brittle-type material at room temperature and low hydrostatic confining pressures, with little or no strain rate sensitivity. At temperatures above about 5500°F , extremely time-dependent behavior is observed, while at moderate hydrostatic pressures, large changes in fracture behavior were seen.

Fig. 1. Schematics of Medium Strain Rate Machine and Hopkinson Bar.

Fig. 2. Medium Strain Rate Machine and Hopkinson Bar Device.

Fig. 3. Composite Stress-Strain Curves for ATJ-S Graphite.

Fig. 4. Schematic of Biaxial Strain Rate Machine.

Fig. 5. Biaxial Strain Rate Machine.

Fig. 6. Biaxial Fracture Stress Surface for ATJ-S Graphite at Room Temperature (Axial Direction Across Grain).

Specimen : 2.50 in. O. D. x 2.00 I. D. x 5.00 in. Gage Length
(Long Axis is Across Grain Direction)

- Compression
- Compression - Tension
- ◇ Compression - Compression
- △ Tension - Tension

Fig. 7. Fracture Strain Surface for ATJ-S Graphite (Axial Direction Across Grain).

Fig. 8. Schematic of High-Temperature Medium Strain-Rate Machine.

Fig. 9. High-Temperature Testing Facility.

Fig. 10. Fracture Stress vs Temperature and Heating Rate for ATJ-S Graphite.

Heated to 6300°F

.010"

Raw Material

.010"

Fig. 11. Photomicrographs of ATJ-S Graphite.

*Material-Design Concepts for Filament-Wound
Graphite-Graphite Heatshields*

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ABSTRACT

Three material-design concepts that may permit a filament-wound graphite-graphite composite (graphite fiber/graphite matrix) heatshield to achieve improved performance during reentry are evaluated. One concept is the use of a constant helical wrap angle, another is the use of the so-called quasi-isotropic wrap, and the final concept is the use of low pressures during the CVD (chemical vapor deposition) process of depositing the matrix. The pressure may be held constant, or it may be varied during the deposition process to achieve property gradients through the shell thickness.

To evaluate the proposed design concepts, calculations were carried out to determine the ratios of the maximum thermal stresses induced by reentry heating to the strength of the composite material. The analysis used laminated orthotropic shell theory and experimentally measured material properties. The calculations showed that only the last concept mentioned results in a better safety margin than the currently used balanced helical ($\pm \theta$) wrap CVD'd at atmospheric pressure.

INTRODUCTION

In the development of heatshields for reentry applications, there is continuing effort to achieve better performance through the use of improved materials and design concepts. With this objective, Sandia Laboratories, Albuquerque, is developing heatshields made of graphite-graphite composites. One design uses carbon felt with a CVD (chemical vapor deposited) carbon matrix¹; another uses balanced-helical-wound carbon fibers with a CVD matrix². These carbon-carbon composites are subsequently heat-treated at high temperatures (approximately 3000°C) resulting in a graphite-graphite composite. Prototypes of both material designs have been test flown successfully.

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The use of composite materials allows the designer freedom to design the material as well as the structural configuration. The present paper describes some concepts that could possibly permit the FW (filament wound)/CVD composite heatshield to achieve improved performance over that of the present design. In the present design of the conical-frustum heatshield structure, the pattern used in the helical filament winding results in a constant wall thickness. To achieve the constant wall thickness along the length of the cone, the angle of wrap ($\pm \theta$) varies between approximately 45° (measured from the shell axis) at the small end to 80° at the large end. Also, in the present design, the CVD carbon matrix is deposited at atmospheric pressure.

The concepts considered for design improvement are (1) the selection of a constant $\pm \theta$ wrapping pattern along the length of the shell, (2) the use of the so-called quasi-isotropic wrap or lamination scheme, and (3) the modification of the deposition pressure during the CVD process to achieve combinations of matrix properties more desirable for reentry heating conditions.

The feasibility of these design concepts are investigated by performing thermal stress analyses of an orthotropic shell in a reentry environment to compare the maximum thermal stresses to the failure strengths of the orthotropic graphite-graphite composite under multi-axial stress conditions. In each of the analyses the temperature distribution through the shell thickness remains the same. The temperature profile used is for the specific time during reentry that the temperature gradient is greatest. During reentry, the temperature distribution obviously changes with time. Thus, in this study, an indication of optimum design may be true only for the time representative of the temperature profile used and not for the entire reentry sequence.

The paper will discuss (1) the method for calculating the thermal stresses resulting from reentry heating, (2) the failure criteria considered, and (3) the material-design concepts considered and their evaluation.

STRESS ANALYSIS

Background

The strength of an orthotropic shell structure subjected to non-linear radial temperature distribution which is typical of reentry, is important in the design and development of filament-wound heatshields. Since it is quite costly to make a complete thermal stress analysis for each trial design or design concept of reentry heatshield being investigated, it is desirable to have a simple method for determining the thermal stresses in a thin-walled shell of revolution with an axisymmetric temperature distribution due to aerodynamic heating. In this section, the history of stress analysis for orthotropic structures is discussed.

Timoshenko and Woinowsky-Krieger³ suggested the following approximate equation for the maximum tension stress, $\sigma_{\max}$, due to a radial temperature gradient distributed uniformly along the length in a circular cylindrical shell of isotropic material:

$$\sigma_{\max} = E \alpha \Delta T / 2 (1 - \nu) \quad (1)$$

where E is Young's modulus, ν is Poisson's ratio, α is the coefficient of lineal thermal expansion, and ΔT is the temperature difference between the outside and inside wall. It should be noted that Eq. (1) is valid only for a linear temperature distribution and at great distances from the ends of the shell.

Bert⁴ investigated the thermal-stress design of isotropic circular cylindrical shells of semi-infinite length with four kinds of end con-

ditions: (1) completely free, (2) completely fixed, (3) simple support (fixed radially; free rotationally), and (4) free radially, fixed rotationally. Cases 1, 3, and 4 gave stresses ranging from 4 to 10% greater than given by Eq. (1); Case 2 gave stresses 240% greater. It was shown that the temperature gradient along the axis of the shell, due to aerodynamic heating, can usually be neglected⁴. Furthermore, by comparison with the thermal-stress calculations of Huth⁵ for a conical shell of small apex angle, it has been shown that the use of an equivalent cylindrical shell is valid (within 4% for the Ref. 5 example).

Although thermoelastic analyses of anisotropic shells have been in existence for a number of years, most of them have been limited to transversely isotropic materials (such as pyrolytic graphite) and to plane-strain elasticity analysis (i.e., neglecting edge effects). One of the earliest analyses within the context of orthotropic thin-wall theory was presented by Heer⁶, but it merely formulated the problem.

Perhaps the first numerical results for thermal-stress edge effects in orthotropic cylindrical shells were presented by Schamaun⁷ for the case of a free edge. The inclusion of end effects in this problem resulted in a 10% increase in maximum tensile stress and a 40% increase in the maximum compressive stress, thus pointing out the importance of using proper boundary conditions.

Theoretical Considerations

The stress analysis developed to evaluate the three proposed design concepts was based on the thermal stress analysis of a free-edge, semi-infinite, specially orthotropic, circular cylindrical shell subjected to an axisymmetric temperature distribution. The equations listed in the Appendix (the details of the derivation are not included)

were derived from thin-shell theory using the equilibrium equations, Love's first-approximation linear strain-displacement relations, and the generalized Duhamel-Neumann thermoelastic relations extended to orthotropic materials. The assumptions inherent in the analysis are as follows:

- H1. The Kirchhoff-Love hypothesis is used, i.e. plane cross sections remain plane, normal to the deflected middle surface, and do not change in thickness. This is the shell version of the Bernoulli-Euler hypothesis used in thin beam theory. Specifically it means that thickness-shear and thickness-normal strains are neglected.
- H2. Displacement and slope changes are assumed to be sufficiently small so that linear expressions for curvature changes and middle-surface strains may be used.
- H3. The shell thickness is uniform and small relative to the radius of the shell.
- H4. The shell material is macroscopically homogeneous, and specially orthotropic^{8,9}. This is a reasonable engineering assumption to make for a filament-wound shell having many layers of wrap, provided that the wrap is "symmetric" with respect to the shell middle surface and "balanced" with respect to the shell axis. All of these requirements are met by the winding schemes investigated, see Figure 1.
- H5. The shell material is linear, perfectly elastic. This assumption considerably simplifies the analysis. At room temperature, the stress-strain data indicates the material is "bi-linear" with some degree of inelasticity. However, Schamaun suggests that at temperatures of 3000°F the stress-strain curve will be linear to failure.¹⁰

H6. The thermal-expansion coefficients are independent of temperature, but the elastic coefficients may vary with temperature (and thus with radial position in the shell).

The neglect of thickness-normal strain due to mechanical loadings is reasonable; however, in view of the high thickness direction thermal expansion coefficient of the present material (see Table 1) this may not be realistic here. Calculations by Schamaun¹¹ using three-dimensional body elasticity codes indicated that this effect is small (<5%).

The equations presented in the Appendix were derived by solving the governing differential equation subject to the free edge boundary condition. Although the equations of Schamaun¹² were derived in an entirely different method (by the semi-inverse method, using superposition), it can be shown that for the case of linear temperature gradient and temperature-independent properties, the equations are equivalent.

STRENGTH ANALYSIS

The apparent integrity of an orthotropic shell structure subjected to a reentry environment depends upon the calculated stresses as compared with the allowable strengths. Just as in the case of "homogeneous" isotropic materials, a number of failure criteria have been proposed to predict the strength of composite materials subjected to multiaxial stresses.

One of the most popular composite failure criteria is the Hill-Tsai¹³ which is widely used for filament-wound glass/epoxy composites. The major disadvantage of this criterion with regard to its application to brittle carbon composites is the assumption that the composite strengths in uniaxial tension and compression are identical. This weakness in the Hill-Tsai criterion was corrected by Hoffman¹⁴.

A deficiency of both the Hill-Tsai and the Hoffman criteria is the fact that they do not permit explicit distinction among various modes of failure. A method of making such a distinction was suggested by Kelly and Davies.¹⁵ In essence, it amounts to adaptation of the maximum-normal-stress and maximum-shear-stress failure theories from isotropic to orthotropic materials. In this criterion the maximum principal stress and maximum shear stress are not significant; instead one must use the following stresses related to the fiber directions: (1) normal stress parallel to fibers (L direction), (2) normal stress transverse to fibers (T direction), (3) shear stress related to L, T axes.

For calculations in this study, only the three individual-failure-mode criteria were considered. Calculated stresses were transformed to the fiber directions and dimensionless stress ratios determined. The stress ratio is defined as the stress divided by the strength. The lower the magnitude of the stress ratio, the better the design. Stress ratios greater than 1.0 indicate failure.

The equations appropriate for each of the strength theories and stress ratios considered are listed in the Appendix, Eqs. (A13).

DESIGN CONCEPTS AND NUMERICAL RESULTS

Helical Wrap Lamination Scheme

The first step in the numerical evaluation of the heatshield design was to check (1) the effect of filament orientation, (2) the effect of the free-edge boundary conditions, and (3) the critical position within the shell structure, i.e. the position in the shell where the stress ratios have the largest values.

The equations presented in the Appendix were used to make the stress ratio calculations for the three individual failure modes criteria. The

material property data used in the calculations are listed in Table 1. These data¹⁰ are the currently used single-ply properties of the CY2-5 filament/CVD matrix (heat-treated at 3000°C) composite. In the calculations the room-temperature strengths are increased according to temperature as shown in Fig. 2; room-temperature moduli are decreased according to temperature as shown in Fig. 2.

The temperature profile used is shown in Fig. 3. It is the same profile used in Reference 12, having a temperature of 4480°F at the outside wall and 1930°F at the inside wall and a nonlinear temperature distribution through the shell thickness. This represents the maximum temperature gradient during reentry heating.

The numerical results for the $\pm \theta$ wrap were determined in the form of stress ratios versus wrap angles at four locations on the shell: inside and outside walls at the free end, and inside and outside walls at a large distance from the free end. The results indicated that the critical location is always in the body of the shell on the inside wall. Figures 4 and 5 show the stress ratios versus wrap angle for the locations in the body of the shell. Since the stress ratios are much lower at the outside wall, the critical location (according to this analysis) is at the inside wall with failure due to tension transverse to the fibers, S_T . As can be seen for the critical case (S_T in Figure 5), the effect of helical angle between 45° and 90° is less than 7 percent and can be considered small in relation to the uncertainty with which the properties (especially the elevated-temperature data) are known. Thus, the concept of a constant wrap angle along the length of the heatshield does not appear promising for improving the heatshield performance.

Quasi-Isotropic Lamination Scheme

Werren and Norris¹⁶ have shown that it is possible to orient the layers of multiple-layer laminates in such a way that the stretching

behavior is isotropic, i.e., has elastic coefficients which are independent of orientation in the plane. Since a laminate made according to the Werren-Norris design (for example, a combination having layers oriented at wrap angles of 30° , 90° , and 150° from the axis) is isotropic in regard to stretching only and not, in general, in regard to bending, the design is called quasi-isotropic. This concept has been previously used in resin-tape layup of flat laminates for multi-axial mechanical loading applications, but apparently not for applications involving thermal stresses.

The only changes in the equations that are required to analyze the quasi-isotropic case is in the transformation of the material or ply properties to the shell principal directions. These are listed in the Appendix as Equations (A-4) and (A-5). Numerical results were obtained for two quasi-isotropic wraps ($30^\circ/90^\circ/150^\circ$ and $0^\circ/45^\circ/90^\circ/135^\circ$) and are shown in Table 2. The room-temperature material property data, the effect of temperature on strength and moduli, and the temperature distribution were the same as used in the $\pm \theta$ wrap calculations. As before, the location of the critical stress ratio was found to be in the body of the shell at the inside surface, and transverse tension was the governing failure mechanism. The 90° direction filaments were critical for both quasi-isotropic wraps. Table 3 compares the results in Figures 4 and 5 with those in Table 2. The comparison indicates that the quasi-isotropic layup is worse than the $\pm \theta$ wrap and thus does not appear promising for the improvement of the heatshield performance.

Gradation of Matrix Properties

Background

In the thermal-stress calculations previously discussed for a FW/CVD carbon composite shell subjected to thermal conditions typical of re-

entry, it was found that the most critical location was always at the inside wall of the composite. Furthermore, the quantities which developed the thermal stresses were the thermal membrane forces (N_{Ti}) and thermal bending moments (M_{Ti}) which are proportional to the following quantities, respectively:

$$\int_{-h/2}^{h/2} \beta_i dZ \quad \text{and} \quad \int_{-h/2}^{h/2} \beta_i Z dZ \quad (i = 1, 2) \quad (2)$$

where h = total wall thickness, Z = thickness coordinate measured outward from the middle surface, and β_i = thermal stress coefficients given by:

$$\beta_1 = Q_{11}\alpha_1 + Q_{12}\alpha_2, \quad \beta_2 = Q_{12}\alpha_1 + Q_{22}\alpha_2 \quad (3)$$

where α_1, α_2 are the axial and hoop thermal expansion coefficients, and the Q 's are material stiffness coefficients (see Appendix).

It is apparent, then, that the magnitude of the thermal stresses can be reduced by reducing the values of the moduli and α 's. In order for the stress ratios to be reduced also, the strengths must not decrease in the same proportion as the moduli and α 's. Also, since the magnitude of the thermal stresses vary with position through the shell thickness, it might be desirable to have strength and moduli decreasing gradually from the inside wall to the outside wall or vice-versa. The concept of having a property gradation* through the thickness is not new in heatshield applications. It has previously been investigated by both LTV Aerospace Corporation¹⁷ and General Electric¹⁸. To achieve property gradation in the FW/CVD carbon composite heatshields, Stoller¹⁹ suggested that it might be practical to vary the pressure gradually during the deposition process. The purpose of the calculations reported here is to investigate the feasibility of this variable-pressure

*A gradual change of properties.

concept as well as constant vacuum pressure on the calculated thermal stresses. Data on the effect of deposition pressures below atmospheric on the mechanical properties of CVD carbon felt were used.

Discussion

Since it is believed that the CVD matrix in the FW/CVD composite may govern the composite behavior²⁰ and that the CVD matrix should respond to pressure during deposition in a similar fashion to CVD carbon felt, data obtained in the carbon-felt investigation were thought to be appropriate.²¹ These results are summarized in Table 4 and plotted in Figures 6-8. No comparative data on the effect of pressure at deposition on the thermal properties were available when the original calculations reported here were performed. Generally a carbon material which has a lower modulus also has a higher thermal expansion coefficient. However, since no quantitative data were available in this instance, it was assumed that the thermal properties are unaffected by pressure at deposition.

Thermal-strength calculations were carried out using the analysis previously described. The same temperature gradient, change in properties with temperature, thermal-expansion coefficients, and major Poisson's ratio were used. The elastic and shear moduli and ply strengths were ratioed from those of Table 1 (which included allowance for the effect of residual stresses on room-temperature strength values) in accordance with Figures 6 and 7.

Four different cases were considered:

1. Case 2 - considering all material to have the same properties due to deposition at 375 mm Hg (the point where the tension and compression curves in Figure 8 cross).
2. Case 3 - considering all material to have the same properties due to deposition at 35 mm Hg.

3. Case 4 - considering elastic and shear moduli and strengths to vary linearly from the inside wall (deposited at atmospheric pressure) to the outside wall (deposited at 35 mm Hg).
4. Case 5 - the reverse of Case B, i.e. linear variation from 35 mm Hg deposition at inside wall to atmospheric pressure deposition at outside wall.

Table 5 gives a complete listing of the room-temperature properties used for the four cases considering either constant or variable vacuum during the CVD process. Based on the results of the earlier calculations, stress ratios were determined at two locations (inside and outside walls in the body of the shell) for two $\pm \theta$ wrap angles (45° and 80°) and for the 90° orientation in the quasi-isotropic wrap. The results that consider variation in the CVD matrix properties are listed in Table 6 along with results for atmospheric pressure deposition (called Case 1, Figures 4 and 5, which are included for comparison purposes).

As can be seen from Table 6, the location of the critical stress ratio is at the inside wall of the shell for Cases 1, 2 and 3, and the indicated failure mechanism is tension transverse to the filaments (S_T). For Cases 4 and 5, the location of the critical stress ratio is the outside wall of the shell and the indicated failure mechanisms are compression normal to the filaments (S_L) for Case 4 and compression transverse to the filaments (S_T) for Case 5.

Since it is desirable to have the dimensionless critical stress ratios (encircled in Table 6) as low as possible, the winding pattern/process combinations that show promise for heatshield improvement are the presently used $\pm \theta$ wrap with the carbon matrix deposited according to Case 3 or Case 5. For uniform deposition at 35 mm Hg (Case 3), the indicated improvement over the presently used material (Case 1) is approximately 60 percent; and the linear gradation scheme of 35 mm Hg

deposition at the inside wall to atmospheric deposition at the outside wall (Case 5) results in an indicated improvement of nearly 100 percent.

SUMMARY AND CONCLUSIONS

Three material-design concepts that may permit a filament-wound graphite-graphite composite heatshield achieve improved performance during reentry have been evaluated. Attention was devoted to the effect of wrap angle in a $\pm \theta$ helical wrap pattern, to two different quasi-isotropic lamination schemes, and to the concept of varying the deposition pressure during the CVD process. The results of the study indicate the following:

1. The effect of wrap angle in the $\pm \theta$ helical wrap pattern is small, thus the selection of a single wrap angle would not significantly improve heatshield performance.
2. The performance of a heatshield having the balanced helical wrap pattern could be improved more than 60 percent by depositing the CVD matrix at a reduced pressure of 35 mm Hg. Even greater improvement is indicated for one case of property gradation through the wall thickness (pressure of CVD deposition varying linearly from 35 mm Hg at the inside wall to atmospheric at the outside wall).
3. The performance of a heatshield is not improved when a quasi-isotropic lamination pattern rather than the balanced helical-wrap pattern is used.

It is emphasized that the above conclusions are based on the data used and the assumptions made in this study. However, the results do indicate that the material design concepts of CVD at pressures below atmospheric and gradation of properties deserve consideration in the future development of filament-wound graphite-graphite heatshields.

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TABLE 1. ROOM TEMPERATURE PLY PROPERTIES

(CY2-5 Filament/CVD Matrix, Heat Treated 3000°C)

<u>Property</u>	<u>Tension</u>	<u>Compression</u>
E_L , psi	4.0×10^6	4.0×10^6
E_T , psi	1.8×10^6	1.8×10^6
G_{LT} , psi	1.0×10^6	1.0×10^6
ν_{LT}	0.52	0.52
F_L , psi	13,000	13,000
F_T , psi	6,000	12,000
F_{LT} , psi	6,000	6,000
α_L , (in/in)/°F	1.7×10^{-6}	1.7×10^{-6}
α_T , (in/in)/°F	3.5×10^{-6}	3.5×10^{-6}

TABLE 2. LARGEST STRESS RATIOS FOR TWO QUASI-ISOTROPIC LAYUPS

<u>Stress Ratios</u>	<u>Three-Directional Layup</u> (30°/90°/150°)		<u>Four-Directional Layup</u> (0°/45°/90°/135°)	
	<u>Inside Wall</u>	<u>Outside Wall</u>	<u>Inside Wall</u>	<u>Outside Wall</u>
S_L	0.27	0.27	0.47	0.15
S_{LT}	0.19	0.12	0.22	0.14
S_T	1.02	1.02	1.02	0.09

TABLE 3. SUMMARY TABLE OF GOVERNING STRESS RATIOS*

<u>Winding Scheme</u>	<u>Maximum Stress Ratio</u> S_T
<u>± θ - Wrap:</u>	
0°	.51
45°	.78
90°	.81
<u>Quasi-Isotropic:</u>	
30°/90°/150°	1.02
0°/45°/90°/135°	1.02

*Largest of S_L , S_{LT} , and S_T at 5 inches from end of shell.

TABLE 4. EFFECT OF PRESSURE DURING DEPOSITION ON PROPERTIES OF CVD CARBON FELT
(Data Summarized from Ref. 21)

Pressure at Deposition mm Hg	Density gm/cm ³	Modulus, Millions of psi			Strength, ksi			% Elong. in 1 in.	
		Tension	Compression	Flexure	Tension	Compression	Flexure	Tension	Compression
35	1.75	2.2	2.0	2.0	4.78	23.5	6.18	0.29	1.4
100	1.79	3.7	2.8	3.0	6.55	35.5	9.07	0.20	1.4
760 (atmospheric)	1.78	4.7	3.3	4.0	6.26	51.8	9.26	0.16	1.73

TABLE 5. ROOM TEMPERATURE PLY PROPERTIES FOR DIFFERENT DEPOSITION PRESSURES

Property	*Case 2		Case 3		Case 4		Case 5	
	Tens.	Comp.	Tens.	Comp.	Tens.	Comp.	Tens.	Comp.
E _L (psi x 10 ⁶)	3.79	3.88	1.87	2.42	4.0	2.42	1.87	4.0
E _T (psi x 10 ⁶)	1.70	1.75	0.84	1.09	1.8	1.09	0.84	1.8
G _{LT} (psi x 10 ⁶)	0.95	0.97	0.47	0.61	1.0	0.61	0.47	1.0
ν _{LT}	0.52	0.52	0.52	0.52	0.52	0.52	0.52	0.52
F _L (ksi)	13	11.5	9.95	5.90	13	5.90	9.95	13
F _T (ksi)	6	10.7	4.60	5.45	6	5.45	4.60	12
F _{LT} (ksi)	6	5.3	4.60	2.72	6	2.72	4.60	6
α _L [(in/in)/°F] x 10 ⁻⁷	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
α _T [(in/in)/°F] x 10 ⁻⁷	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5

*Case 2 - Uniform deposition at 375 mm Hg.

Case 3 - Uniform deposition at 35 mm Hg.

Case 4 - Linear variation, atmospheric pressure deposition at inside wall to 35 mm Hg deposition at the outside wall.

Case 5 - Reverse of Case 4.

TABLE 6. STRESS RATIOS FOR THE FIVE CASES CONSIDERED

(The critical stress ratio for each case is encircled)

STRESS RATIO	*Case 1		Case 2		Case 3		Case 4		Case 5	
	Inside Wall	Outside Wall	Inside Wall	Outside Wall	Inside Wall	Outside Wall	Inside Wall	Outside Wall	Inside Wall	Outside Wall
<u>Helically Wrapped at $\pm 45^\circ$</u>										
S_L	0.36	0.08	0.34	0.08	0.22	0.10	0.09	0.72	0.11	0.34
S_T	0.78	0.08	0.74	0.09	0.47	0.06	0.20	0.39	0.24	0.37
S_{LT}	0.27	0.15	0.22	0.17	0.14	0.20	0.03	0.03	0.23	0.25
<u>Helically Wrapped at $\pm 80^\circ$</u>										
S_L	0.24	0.00	0.22	0.00	0.14	0.00	0.07	0.74	0.24	0.22
S_T	0.81	0.12	0.77	0.14	0.49	0.08	0.19	0.31	0.02	0.40
S_{LT}	0.05	0.05	0.05	0.05	0.03	0.06	0.05	0.07	0.09	0.06
<u>Quasi-Isotropic Wrap (90° Direction)</u>										
S_L	0.27	0.02	0.25	0.02	0.16	0.02	0.08	0.73	0.20	0.25
S_T	1.02	0.16	0.96	0.17	0.61	0.10	0.23	0.38	0.01	0.50
S_{LT}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

*Case 1 - Uniform deposition at atmospheric pressure.

Case 2 - Uniform deposition at 375 mm Hg.

Case 3 - Uniform deposition at 35 mm Hg.

Case 4 - Linear variation, atmospheric deposition at inside wall to 35 mm Hg deposition at the outside wall.

Case 5 - Reverse of Case 4.

Fig. 1. Winding Schemes Investigated.

Fig. 2. Assumed Effect of Temperature on Mechanical Properties.

Fig. 3. Assumed Temperature Profile Through the Thickness of the Shell.

Fig. 4. Stress Ratios vs Wrap Angle for a Position in the Body of the Shell at the Outside Wall.

Fig. 5. Stress Ratios vs Wrap Angle for a Position in the Body of the Shell at the Inside Wall.

Fig. 6. Effect of Pressure on Modulus.

Fig. 7. Effect of Pressure on Strength and Ductility.

Fig. 8. Effect of Pressure on Strength-to-Modulus Ratio.

APPENDIX

This appendix lists the equations required for the thermal stress analysis of a free-edge, semi-infinite, specially orthotropic, circular cylindrical shell subjected to an axisymmetric temperature distribution which is uniform along the length. The in-surface shell coordinate axes (designated as 1-axial direction and 2-hoop direction are referenced on the middle surface of the shell wall. The material coordinate axes (L-T) are parallel and transverse to the filament directions. The angle θ is measured between the 1-axis and the L-axis.

NOMENCLATURE

E_L, E_T (psi)	Single-ply elastic moduli parallel & transverse to fibers
F_L, F_{LT} (psi)	Single-ply strengths parallel to fibers and in shear
$F_T^{(-)}, F_T^{(+)}$ (psi)	Single-ply compressive & tension strengths transverse to fibers
G_{LT} (psi)	Single-ply in-plane shear modulus relative to fiber axes
h (inches)	Shell thickness
M	$\cos^2 \theta$
R (inches)	Shell radius
Q_L, Q_{LT}, Q_S, Q_T (psi)	Single-ply planar elastic coefficients relative to fiber axes
Q_{11}, Q_{12}, Q_{22} (psi)	Planar elastic coefficients of the composite relative to shell axes
S_L, S_T	Stress ratios for normal-stress criteria (parallel and transverse to fibers)
S_{LT}	Stress ratio for shear-stress criterion
T_i, T_o ($^{\circ}\text{F}$)	Inside and outside wall temperatures
$T_m, \Delta T$ ($^{\circ}\text{F}$)	Effective mean temperature and temperature gradient

R.T.	Room Temperature
x (inches)	Axial coordinate, measured from the free end
Z (inches)	Thickness coordinate, measured positive outward from mean radius
α_L, α_T (in/in/°F)	Single-ply thermal-expansion coefficient parallel and transverse to fibers
α_1, α_2 (in/in/°F)	Thermal-expansion coefficients of the composite relative to shell
β_1, β_2 (psi/°F)	Thermal-stress coefficients of the composite relative to shell axes
$\beta_1^{(i)}, \beta_1^{(o)}$ (psi/°F)	β_1 at inside and outside walls
θ (degrees)	Angle between fibers and shell axis
λ	$1 - \nu_{LT} \nu_{TL}$
ν_{21}	Minor Poisson's ratio of the composite relative to shell axes
σ_L, σ_T (psi)	Normal stresses parallel and transverse to fibers
σ_{LT} (psi)	In-plane shear stress relative to fiber axes
$\sigma_1^{(i)}, \sigma_2^{(i)}$ (psi)	Axial and hoop stresses at inside wall
$\sigma_1^{(o)}, \sigma_2^{(o)}$ (psi)	Axial and hoop stresses at outside wall

Single Ply Stiffness Coefficients

$$\begin{aligned}
 Q_L &= E_L / \lambda \\
 Q_T &= E_T / \lambda \\
 Q_{LT} &= \nu_{TL} E_L / \lambda \\
 Q_S &= G_{LT}
 \end{aligned}
 \tag{A1}$$

Shell Principal Directions (1,2) Stiffness Coefficients For Helical ($\pm \theta$) Wrap

Elastic Properties:

$$\begin{aligned}
 Q_{11} &= Q_L M^2 + (2Q_{LT} + 4Q_S) M (1-M) + Q_T (1-M)^2 \\
 Q_{12} &= Q_{LT} M^2 + (Q_L + Q_T - 4Q_S) M (1-M) + Q_{LT} (1-M)^2 \\
 Q_{22} &= Q_T M^2 + (2Q_{LT} + 4Q_S) M (1-M) + Q_L (1-M)^2
 \end{aligned}
 \tag{A2}$$

Thermoelastic Properties:

$$\begin{aligned} \beta_1 &\equiv Q_{11}\alpha_1 + Q_{12}\alpha_2, \quad \beta_2 \equiv Q_{12}\alpha_1 + Q_{22}\alpha_2 \\ \text{where} \\ \alpha_1 &= \alpha_L M + \alpha_T (1-M), \quad \alpha_2 = \alpha_L (1-M) + \alpha_T M \end{aligned} \quad (A3)$$

For Quasi-Isotropic Wrap

Elastic Properties:

$$\begin{aligned} Q_{11} &= Q_{22} = (1/8) [3(Q_L + Q_T) + 2 Q_{LT} + 4 Q_S] \\ Q_{12} &= (1/8) (Q_L + Q_T + 6 Q_{LT} - 4 Q_S) \end{aligned} \quad (A4)$$

Thermoelastic Properties:

$$\begin{aligned} \beta_1 &\equiv Q_{11} \alpha_1 + Q_{12} \alpha_2, \quad \beta_2 = Q_{12} \alpha_1 + Q_{22} \alpha_2 \\ \alpha_1 &= \alpha_2 = (1/2) (\alpha_L + \alpha_T) \end{aligned} \quad (A5)$$

TEMPERATURE VALUES

Outside Wall: $T_o = 4480^\circ\text{F}$

Inside Wall: $T_i = 1930^\circ\text{F}$

$$\text{Effective mean temperature: } T_m = \left(\frac{1}{h}\right) \int_{-\frac{h}{2}}^{\frac{h}{2}} \frac{\beta(Z)}{\beta_{R.T.}} T(Z) dZ \quad (A6)$$

$$\text{Effective temperature gradient: } \Delta T = (12/h^2) \int_{-h/2}^{h/2} \frac{\beta(Z)}{\beta_{R.T.}} T(Z) Z dZ \quad (A7)$$

STRESSES WITH RESPECT TO SHELL AXES

Outside Wall

$$\begin{aligned} \text{Axial: } \sigma_1^{(o)} &= \frac{1}{2} \beta_1 \Delta T \phi + \beta_1 T_m - \beta_1^{(o)} T_o \\ \text{Hoop: } \sigma_2^{(o)} &= \left\{ -[(Q_{22}Q_{11}^{-1} - (Q_{12}/Q_{11})^2) (1/3)]^{\frac{1}{2}} \psi + \right. \\ &\quad \left. (Q_{12}/Q_{11}) \right\} (\beta_1 \Delta T / 2) + (\beta_2 / \beta_1) (\beta_1 T_m - \beta_1^{(o)} T_o) \end{aligned} \quad (A8)$$

where

$$\phi, \psi = e^{-\delta x} (\cos \delta x \pm \sin \delta x) \quad (A9)$$

and

$$\delta = (hR)^{-\frac{1}{2}} [3(Q_{22}Q_{11}^{-1} - (Q_{12}/Q_{11})^2)]^{\frac{1}{4}} \quad (A10)$$

Inside Wall

$$\begin{aligned} \text{Axial: } \sigma_1^{(i)} &= -\frac{1}{2} \beta_1 \Delta T \phi + \beta_1 T_m - \beta_1^{(i)} T_i \\ \text{Hoop: } \sigma_2^{(i)} &= \left\{ -[(Q_{22}Q_{11}^{-1} - (Q_{12}/Q_{11})^2) (1/3)]^{\frac{1}{2}} \psi \right. \\ &\quad \left. - (Q_{12}/Q_{11}) \right\} \beta_1 \Delta T / 2 + (\beta_2/\beta_1) (\beta_1 T_m - \beta_1^{(i)} T_i) \end{aligned} \quad (A11)$$

TRANSFORMATION OF STRESSES TO FIBER DIRECTIONS

$$\begin{aligned} \sigma_L &= (1/2) [(\sigma_1 + \sigma_2) + (\sigma_1 - \sigma_2) (2M-1)] \\ \sigma_T &= (1/2) [(\sigma_1 + \sigma_2) - (\sigma_1 - \sigma_2) (2M-1)] \\ \sigma_{LT} &= (\sigma_1 - \sigma_2) [M(M-1)]^{\frac{1}{2}} \end{aligned} \quad (A12)$$

STRESS RATIOS (FAILURE CRITERIA)

Maximum Normal Stresses

$$\text{Parallel with fibers: } S_L = \sigma_L / F_L$$

$$\begin{aligned} \text{Transverse to fibers: } S_T &= \begin{cases} \sigma_T / F_T^{(+)} & \text{if } \sigma_T > 0 \text{ (Tension)} \\ \sigma_T / F_T^{(-)} & \text{if } \sigma_T < 0 \text{ (Compression)} \end{cases} \end{aligned} \quad (A13)$$

Maximum Shear Stress

$$S_{LT} = \sigma_{LT} / F_{LT}$$

THE MICROSTRUCTURAL EVALUATION OF GRAPHITES
FOR THEIR EROSION AND ABLATION CHARACTERISTICS
IN AEROSPACE APPLICATIONS

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ABSTRACT

A program has been undertaken to characterize the microstructures of graphites and their potential susceptibility to erosion and ablation. The pore structure of the graphites is considered to be a primary factor in determining the mass loss of carbon. Therefore, a combination of tests has been used to define the pore structure and those factors that influence it. These tests include optical microscopy for grain and macropore size determination; scanning electron microscopy for microporosity and binder/filler distribution; and gas permeability, helium density, and surface area determinations for apparent pore size distribution. Different types of graphites have been subjected to short-term oxidation and sublimation as a preliminary base toward obtaining a better understanding of the influence of microstructure. Nine types of graphites were selected, with different grain sizes, pore distributions, and types of fillers and binders, to determine the effect of the test conditions.

This combination of tests has revealed several trends in the microstructure that appear to be influential. The pore structure is composed of both micropores and macropores. The micropores are a result of the usual shrinkage caused during pyrolysis of the binder phase; the macropores are formed by the packing of the grain or flour. A second factor in determining the microporosity appears to be the agglomeration of filler and binder into high-density regions 75 to 250 microns in diameter. These are surrounded by lower-density regions which appear as high-gas-permeability zones that may have an influence on the microstructure ablation effects just below the surface of the graphite. The effect of these structures is discussed in detail.

This paper discusses some of the work now being performed under a contract with the Air Force on mechanisms and parameters that may influence the mass loss of graphites under severe reentry conditions. As depicted in Fig. 1, in some of the ground arc tests there was evidence of flaking and of selective etching. Apparently coke particles are being etched out. Therefore, the microstructures of a number of graphites

are being evaluated in an effort to determine whether it is possible, by understanding some of the parameters that influence this, to better understand the mass loss that cannot be fully explained on a purely chemical basis. The general approach has been examination of different microstructures of graphite materials obtained from many sources, characterization of these materials in detail where possible, controlled partial sublimation of specimens of the characterized material, and post-sublimation characterization of some of the structures. The next approach in the program will be more sophisticated ablation and post-ablation tests. This portion of the contract is currently under way.

The characterization tests performed are shown in Fig. 2. In general, the pore structure and some of the factors that relate to the pore structure were examined, concentrating a great deal on gas permeability, scanning electron microscopy, and metallography, as well as some of the density measurements. The graphites supplied come from many sources, including LASL, Lockheed (glassy carbons), Speer, Union Carbide, Great Lakes, and POCO. Also, some graphites were made in our own laboratories, in an endeavor to produce typical kinds of binder-filler ratios or to cover some structural aspects that are not normally produced commercially. The purpose was to use different structures to provide insight on the mechanisms involved during sublimation.

From the scanning electron microscopy, as will be shown below, there seem to be several types of pores, which are depicted by the simplified model in Fig. 3. The large pores are formed by stacking of the idealized flake particles; the smaller pores or fissures in the binder phase, some open and some closed, are certainly a result of some of the conditions during pyrolysis.

This model was used to correlate pore size distributions with ratios of dimensions of the particles, and to examine the variations from graphite to graphite. Figure 4 depicts generally the macropore and micropore types of porosity. The micropores are roughly less than a micron in size; these are undoubtedly caused by shrinkage of the binder that one can envision occurring during pyrolysis. With that sort of concept, some characterization on pore structure was undertaken. Three types of methods for trying to assess the pore volume and pore structure are shown in

Fig. 5. Comparison of pore diameters as observed by mercury porosimetry, by metallography, by BET internal surface area methods in the smaller region, and then by helium density indicates that there are small pores that may well be the entrances to larger pores.

An interesting phenomenon, previously observed and reported by Battelle Memorial Institute, was observed repeatedly in these tests. At some critical pressure during the mercury porosimetry measurements, there were large increases in internal surface area in the smaller size ranges shown in Fig. 5. It was found that there were variations of this critical pressure from graphite to graphite. The critical pressure is related to the mercury pressure or pore size when the surface area becomes larger than was measured by the helium density method.

Another major area that was examined in these various graphites was the gas permeability, shown in Fig. 6 for ATJ-S as a function of sample thickness. The viscous or viscosity term α is indicated by the intercept, and the inertial term β is indicated by the slope. There were wide variations in permeability as sample thickness was varied; this showed that near the surface of the graphite there are nonlinear variations in permeability, which must be considered when one is thinking in terms of carbon leaving graphite during sublimation.

Permeability also varies with the type of graphite. One interesting illustration of this is the comparison between ATJ-S (Fig. 6) and POCO (Fig. 7), which has a finer pore structure. Again, there were variations with total thickness of sample, but this curve for POCO is a different type of permeability, which involves molecular interactions and is more a function of the internal surface area of the pores. The variation with total sample thickness was interpreted as local or extended variations within the microstructure. Sample thickness is only a measure of the extent of the effect.

Nine different types of graphites were examined in an effort to find correlations of these two terms, α and β , with sample thickness or depth below the surface and with microstructure of the various graphites. A fairly good correlation was found, as shown for the viscous term, α , in Fig. 8. Normalization with respect to a thickness indicated that as depth below the surface increases, there is variation in the viscous

resistance term, until at some depth it seems to become uniform. This applied to these various graphites, the mesophase from Great Lakes, AXF-5Q from POCO, ATJ-S from Union Carbide, and some of the MDAC graphites in which we altered the pore structure considerably. As shown in Fig. 9, this correlation held for the inertial term, β , for the same spectrum of graphites as a function of thickness.

After the characterization of the pore structures, sublimation tests were performed in equipment that is schematically shown in Fig. 10. It seems like a very simple problem to heat a disc of graphite to 3000°C at <600 micron pressure. It was difficult, however, because carbon is really emitted at these conditions. Particulate emission was reduced by controlling the pressure, and a tantalum carbide shield was placed around the discs to prevent mass transport between the sample and the graphite susceptor or heater.

These discs were roughly 1-1/4 in. in diameter and 1/4-in. thick. Figure 11 shows the characterization tests that were conducted before and after the test and generally how the samples were cut into sections for evaluation. In order to determine whether there is a change in pore volume in the first few mils below the surface and the extent of that change, there was developed a technique of impregnating with beeswax, microtoming off thin layers, collecting the shavings, reheating and effectively pyrolyzing the beeswax, and then weighing the difference. Thus the term Beeswax Impregnated Microtomed and Pyrolysis (BIMP) test. The results of such a test are shown in Fig. 12, where a volume effect was observed to about 200 microns depth below the sublimed surface. Generally, there is a volume change, and it is a way of integrating the effect. Metallographically and with a scanning electron microscope it was very difficult to see any volume changes.

As mentioned by S. G. Babcock and W. T. Barry in the discussion of the paper by Babcock and his colleagues (earlier in this session of the conference), we found that on heating to high temperatures there was a critical pressure at which particles were evolved like a snowstorm, if the internal carbon pressure within pores was not to some degree balanced by the external system pressure. This may be an important factor in determining mass loss during reentry.

To show the influence of the structure at the surface, scanning electron micrographs were taken of ATJ-S, AXF-5Q, some of the MDAC graphite, and the LASL graphite, as shown in Fig. 13. Note the difference in the magnification. There was a finer structure on the surfaces in some of these graphites. Generally they looked fairly uniform, but a very fine, almost granular type of structure was observed. In some cases the pores were somewhat filled. In MDAC 238 graphite there were not very many pores in evidence, and that was true for this graphite before the sublimation test. On the other hand, the ATJ-S and AXF-5Q graphites showed a fair increase between the pore sizes before and after the sublimation test.

As shown in Fig. 14, there is a correlation between the mass loss and the measured surface area for these sublimations. That is the main point of this work to date; no other very strong correlations have been found for the physical parameters measured and mass loss due to sublimation.

Several interim conclusions have been drawn. Sublimation mass loss does correlate with internal surface area. The molecular permeability seems to vary with internal surface area. The surface roughness seen on sublimation does seem to have some correlation with the macropore major diameters. The increase in internal surface area at some critical pressure during mercury permeability tests is believed to be evidence of weakening of the binder. High pressure data in some of the arc tests showed a dependence of the initial rate on material characteristics. The next step in this program is ablation tests with not only high temperature, but of course high pressures and gas flow. The information derived so far will be related to the larger variations in the microstructure.¹

1. Since the conference an annual report has been published: AFML-TR-70-307, Vol. I, January 1971.

BILLET-FRACTURED

1,000 X

ATJ-S

SHAPED-FRACTURED

1,000 X

BILLET AFTER
ENVIRONMENTAL TEST

1,000 X

BILLET AFTER
ENVIRONMENTAL TEST

1 00 X

Fig. 1. Microstructure (SEM): Shaped vs Billet. Reduced 30%.

1. SURFACE ROUGHNESS
2. PORE SIZE DISTRIBUTION
3. BULK DENSITY OF GRAPHITE OR COMPOSITE
4. SUBMERGED DENSITY OF GRAPHITE, COMPOSITE, FILLER AND BINDER
- 5A. SAMPLE POLISHING AND LIGHT MICROSCOPY PICTURES (2 SPECIMENS/MOUNT)
- 5B. ELECTRON SCANNING MICROSCOPE AND 4-5 PICTURES
- 5C. X-RAY FOR GROSS ORIENTATION
6. MICROSTRUCTURAL EVALUATION FOR:
 - FILLER SIZE AND STRUCTURE
 - COMPOSITE AND GRAPHITE FILLER TO BINDER RATIO
 - FILLER ORIENTATION
 - DISTRIBUTION OF FILLER, BINDER
 - ELECTRON SCANNING FOR BINDER/FILLER INTERFACIAL CHARACTERIZATION
7. MICROHARDNESS
8. GAS PERMEABILITY
9. EVALUATION OF DATA

Fig. 2. Material Characterization Tests.

ATJ-S(S)	$\bar{L}(\mu)$	$\bar{t}_p(\mu)$
	70 (EST.)	20 (EST.)
MDAC-228	16	2

$$8t_B^3 + 2(3\bar{L} + 2t_p)t_B^2 + t_B(\bar{L}^2 + 3\bar{L}t_p)$$

$$\cdot \left(\frac{K_B}{\rho_t} + v_{f_o} + v_{f_c} \right) \frac{\rho_t t_p \bar{L}^2}{2(1 - K_B)}$$

$$1 - \frac{S_c}{S} = \frac{3V_v d_v^2 \rho_t t_p \bar{L}^2}{(1 - K_B) d_v^3} \times$$

$$\left[\bar{L}^2 + 12\bar{L}t_B + 24t_B^2 + t_p(3\bar{L} + 8t_B) \right]^{-1}$$

Fig. 3. Flake Particle Model.

MDAC 3
2000X

MDAC 7
2000X

Fig. 4. SEM Microstructure: Porosity of Control Samples. Reduced 20%.

Fig. 5. Pore Volume Distributions for ATJ-S.

Fig. 6. Permeability of ATJ-S.

Fig. 7. Permeability of AXF-5Q.

Fig. 8. Viscous Resistance Coefficient Correlation.

Fig. 9. Inertial Resistance Coefficient Correlation.

Fig. 10. Schematic of Furnace for Sublimation.

PRETEST

- WEIGHT
- DIMENSIONS
- DENSITY
- GAS PERMEABILITY (CONTROL)
- POROSIMETRY (CONTROL)
- SURFACE AREA (CONTROL)

SAMPLE SHAPE

POST TEST

- WEIGHT
- DIMENSIONS
- MICROSCOPY
- SCANNING ELECTRON
- BIMP
- PYROLYSIS
- GAS PERMEABILITY (<.050" THICK)
- MERCURY POROSIMETRY

Fig. 11. Characterization Sequence (After Sublimation Test).

Fig. 12. Sub-Surface Volume, BIMP Test.

Fig. 13. Surface Structure of Sublimed Graphites. Reduced 30%.

Fig. 14. Correlation of Sublimation Mass Loss with Internal Surface Area of Graphite.

X CHARACTERIZATION OF PRODUCTION-GRADE
H-327 GRAPHITE FOR HTGR DESIGN

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ABSTRACT

The moderator graphite in High-Temperature Gas-Cooled Reactors (HTGRs) is also a containment and structural member of the core. The current core design uses an electrode-type anisotropic production-grade graphite, and the variations in the product that may occur in the production of the required kiloton quantities for HTGRs need to be well established. Knowledge of the behavior of the graphite with respect to mechanical strength, density, permeability, and chemical and thermophysical properties before and after nuclear irradiation is required to permit predictions as to the design and operating performance.

A Great Lakes Carbon Company (GLCC) grade H-327 needle-coke graphite has been selected as the fuel and reflector block material for the Fort St. Vrain HTGR. Measurements of mechanical strength and its variations throughout the graphite log have been made on a portion of the logs produced to date and are correlated with production inspection tests. The strength (at room temperature) in the weakest section of an extruded log gives a safety factor of 3 over stresses resulting from the most pessimistic design assumptions. The strength is increased as a result of irradiation and at Fort St. Vrain median fluence levels ($\sim 5 \times 10^{21}$ n/cm², $E > 0.18$ MeV), the minimum increase in strength is 20%.

The above improvement in strength due to irradiation, together with changes in thermophysical properties and dimensions within a Fort St. Vrain fuel block, has been established in a series of capsule irradiations, at fluences beyond the maximum Fort St. Vrain fast neutron fluence of 8×10^{21} n/cm² ($E > 0.18$ MeV) at temperatures up to and beyond the Fort St. Vrain maximum of 1150°C. It was shown that the rate of change in linear dimensions of H-327 graphite is similar to that of other needle-coke graphites at any given temperature, with slightly greater contraction being experienced before the onset of expansion. These tests also confirm the expected reductions in thermal conductivity of up to $\sim 50\%$ and only slight variation in the coefficient of thermal expansion in the Fort St. Vrain core graphite at the end of life.

These studies have led to a good understanding of the properties and irradiation behavior of H-327 graphite and have shown that it will meet the performance requirements of the Fort St. Vrain HTGR.

INTRODUCTION

The 330-MW(e) Fort St. Vrain High-Temperature Gas-Cooled Reactor (HTGR) being built by Gulf General Atomic is an extension of the basic technology demonstrated in the 40-MW(e) Peach Bottom HTGR. The active core of the Fort St. Vrain reactor is approximately a right cylinder, 19.5 ft in diameter and 15.6 ft high, comprising 1482 graphite fuel element blocks. Each fuel block contains the nuclear fuel, in 1/2-in.-diameter cylindrical rod form, located within 210 columns, which are interspersed with 108 coolant hole channels. Fifteen percent of the graphite fuel elements also contain large through holes for reactor control rod and reserve shutdown systems. The extensive machining required to produce Fort St. Vrain fuel element blocks from a graphite log is shown in Fig. 1.

Fast neutron irradiation and local temperature gradients around the fuel and coolant holes can induce local stresses in the Fort St. Vrain graphite fuel elements. These ligament stresses are generally tensile adjacent to the fuel holes and compressive adjacent to the coolant holes (Fig. 2). The stress is higher at reactor shutdown because a thermal stress component becomes additive to the contraction stress at that time.

PROPERTIES

A nuclear-grade extruded needle-coke graphite (Great Lakes Carbon Company grade H-327) is being used for the Fort St. Vrain fuel elements and replaceable reflector blocks. This graphite has anisotropic characteristics in directions parallel and perpendicular to the extrusion axis. The parallel axis of the log is coincident with the axis of the fuel columns, which are aligned vertically in the reactor core.

The principal physical and chemical requirements for H-327 graphite as specified for the fuel block material are listed in Table 1. These requirements are based on the need for nuclear purity in a well-graphitized material possessing adequate strength. Tensile strength tests are made on samples machined from every log used in the core; the results of the first 541 tests are shown in Fig. 3. To ensure meeting the requirement that all fuel element graphite is subjected to graphitization temperatures of at least 2700°C, the whole log electrical resistivity of each graphite piece is measured. Cline¹ has shown an approximately linear correlation of electrical resistivity with respect to graphitization temperature over the specified temperature range of interest (Fig. 4). The physical and chemical property variations of H-327 graphite logs (Table 2) are shown to be within acceptable levels for fuel element or reflector graphites.

PROPERTY VARIATIONS WITHIN LOGS

In addition to coring for 100% tensile testing, approximately 1% of the graphite logs currently manufactured for Fort St. Vrain have been sectioned and tested for physical, thermophysical, and chemical purity variations within each log. Variations in the density, the preferred orientation (as measured by the Bacon anisotropy factor), and the coefficient of thermal expansion within an extruded log are shown in Table 3.

Typical maximum differences in room temperature uniaxial tensile stress-strain plots for samples at two positions within a log are shown in Fig. 5. The chord modulus at between 250 and 500 psi stress is one of the preferred tensile test parameters, since the stress range covers the expected service range of the fuel element graphite. Tensile property variations are found to

be greatest across the section of an H-327 graphite log at the end and mid-length positions; measured values are given in Table 4.

The measured amounts of nuclear parasitic materials in the production graphite have remained well below specified acceptable levels (Tables 5 and 6) and approximate to less than 1 ppm boron equivalent.

From a series of helium flow measurements on H-327 graphite samples machined from various positions within the log, values for the pore size and the number of pores have been derived. The permeability varies by a factor of four (maximum) for samples within a log and up to seventeen for samples from different logs (Tables 7 and 8).

FAST NEUTRON IRRADIATION EFFECTS

A small portion of the graphite within the Fort St. Vrain core will undergo service temperatures of 1150°C, and the maximum fast neutron exposure will be 8×10^{21} n/cm² ($E > 0.18$ MeV). The bulk of the material will be at temperatures of 700° to 900°C and the median fast fluence will be 5×10^{21} n/cm² ($E > 0.18$ MeV) (Fig. 6). Pilot and production-grade H-327 graphites have been irradiated in a series of capsules; dimensional change plots have established the critical parameters. One of the most critical aspects of the service performance of Fort St. Vrain graphites is the avoidance of any net expansion across the radial direction of the core. As can be seen from Fig. 6, only a small portion (shaded area) of the flux and temperature environment of the Fort St. Vrain core approaches this condition.

The dimensional change behavior of H-327 graphite under fast neutron irradiation is similar to that of other needle-coke materials. In the transverse to extrusion direction (core radial direction), the material contracts, goes through a turnaround, and then expands rapidly. In the direction parallel to extrusion, the graphite contracts at an increasing and then decreasing rate, passes through turnaround, and then begins to expand. The production-grade H-327 graphite is shown to be similar to pilot-grade material in its irradiated dimensional change behavior (Fig. 7) and, in comparison with an isotropic Gilsonite graphite (Figs. 8 through 10), exhibits smaller contraction in the core radial direction. This factor provides a distinct advantage with the present Fort St. Vrain design, although the Gilsonite graphite gives larger margins of safety with respect to crossover (i.e., net expansion).

The effects of irradiation on the mechanical properties of H-327 graphite are similar to other graphite materials in that the strength increases with fluence up to Fort St. Vrain median fluence levels, and the increases are less at higher irradiation temperatures. Increases in tensile properties of H-327 graphite (Fig. 11) at median fluence levels after irradiation at 690° to 1050°C range between 50 and 150% (Table 9).

Fast neutron irradiation of H-327 graphite produces a steep reduction in thermal conductivity at fluences much lower than Fort St. Vrain median fluence levels. Over the service fluence and temperature conditions for Fort St. Vrain, the thermal conductivity of H-327 graphite across the web of a fuel element (i.e., transverse to extrusion) will be reduced by 60% at the lower service temperatures and 40% at close to the peak operating temperatures (Fig. 12). Preliminary analysis shows that the coefficient of thermal expansion is reduced with median Fort St. Vrain fluence levels and peak service temperatures and increases slightly at the lower range of temperature.

CONCLUSIONS

The characterization of production-grade H-327 graphite for HTGR application has established its behavior with respect to strength, density, permeability, chemical and thermophysical properties, and changes due to fast neutron irradiation. The work has shown that a nominally proprietary electrode-type graphite is favorable for the present HTGR concept.

ACKNOWLEDGMENT

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REFERENCE

1. A. A. Cline, Great Lakes Carbon Company, Private Communication, March 13, 1969.
2. G. B. Engle and A. L. Pitner, High-Temperature Irradiation Behavior of Production-Grade Nuclear Graphites, GA-9973 (July 10, 1970).

Table 1. Physical and Chemical Specifications for H-327 Graphite

Bulk Density, g/cm ³	1.72 Min. Avg.
Elect. Resistivity, ohm-inches () ^a	0.00024 Max.
Thermal Conductivity, BTU/hr-ft-°F @ 2200°F (⊥) ^b	18 Desired Min.
Tensile Strength, psi (- room temp.)	1000 Min.
Total Ash, ppm	1000 Max.
Iron, ppm	100 Max.
Titanium, ppm	50 Max.
Vanadium, ppm	50 Max.
Boron, ppm	5 Max.

^a(||) - parallel to extrusion direction.

^b(⊥) - perpendicular to extrusion direction.

Table 2. Physical and Chemical Property Variations of H-327 Graphite

	$\bar{x}$	σ	N	Max.	Min.	Range
Bulk Density, g/cm ³	1.78	0.014	867	1.83	1.74	0.09
Resistivity (II), ohm-inches	0.00017	0.0000087	814	0.00021	0.00015	0.00006
Tensile Strength (II), psi	1667	347	522	2780	1000	1780
Total Ash, ppm	56.10	53.88	94	408	16	392
Iron, ppm	9.92	16.37	70	80.0	1.0	79.9
Titanium, ppm	4.35	7.72	68	33.4	0.2	33.2
Vanadium, ppm	1.22	1.54	81	8.7	0.1	8.6
Boron, ppm	0.50	0.48	145	4.6	0.1	4.5

Table 3. H-327 Production Graphite Uniformity

Log 42/1960

	A	B	C	D
Density, g/cm ³	1.78	1.80	1.74	1.77
Preferred Orientation	2.06	2.21	2.1	1.96
Coefficient of Thermal Expansion (CTE), Mean (I)	3.29	3.45	3.32	3.11
Value between 22°C and 1000°C, 10 ⁻⁶ (II)	1.60	1.56	1.58	1.59

Table 4. Tensile Test Data for Fort St. Vrain Fuel Block Grade H-327 Graphite

Log Position	UTS (psi)	Strain at Fracture (10^{-3} in./in.)	Moduli x 10^{-6} (psi)	
			Secant	Chord
End edge (II)	2300	1.94	1.26	1.71
Mid-length edge (II)	2395	2.11	1.23	1.74
Mid-length center (II)	1630	1.48	1.06	1.54
End center (II)	2180	1.92	0.98	1.51
End edge (I)	1400	2.96	0.56	0.66
Mid-length edge (I)	1290	2.74	0.48	0.65
Mid-length center (I)	920	2.08	0.46	0.58
End center (I)	1349	2.87	0.48	0.68

Table 5. Spectrochemical Analysis of Impurity Levels in Twelve Logs of Fort St. Vrain Grade H-327 Graphite

Log No.	Log Position	Element Concentration (ppm) ^a									Log Avg Density (g/cm ³)
		Al	B	Cu	Fe	Mg	Si	Ti	V	Ash ^c	
694	End edge	1.0	b	--	--	2.0	<10.0	4.0	b	21	1.78
705	End edge	1.0	1.0	--	--	2.0	<10.0	8.0	--	38	1.77
739	End edge	1.0	2.0	--	--	2.0	10.0	10.0	--	55	1.78
744	End edge	4.0	2.0	--	2.0	6.0	20.0	8.0	--	77	1.78
769	End edge	1.0	1.0	1.0	1.0	4.0	20.0	10.0	--	55	1.75
956	End edge	4.0	1.0	1.0	2.0	4.0	20.0	10.0	--	472	1.77
958	End edge	--	1.0	--	2.0	1.0	20.0	20.0	--	317	1.80
1058	End edge	1.0	1.0	--	--	4.0	20.0	10.0	--	72	1.78
1959	End edge	6.0	--	1.0	10.0	10.0	80.0	10.0	--	77	1.77
2479	End edge	4.0	1.0	1.0	1.0	6.0	20.0	10.0	--	65	1.77
2482	End edge	2.0	1.0	--	2.0	6.0	20.0	10.0	--	62	1.78
42/1960	End edge	20.0	10.0	<1.0	2.0	2.0	<10.0	6.0	--	47	
40/9977C	End center	2.0	--	--	1.0	2.0	<10.0	6.0	--		1.77
12-1	Mid-len. edge	4.0	4.0	--	1.0	4.0	20.0	8.0	--		
	Mid-len. ctr.	1.0	--	--	1.0	4.0	10.0				

^a Accuracy 50%.

^b Not detectable below 0.5 ppm.

^c Vendors' reported chemical analysis.

Table 6. Typical Rare Earth and Alkali Metal Contents
of Fort St. Vrain H-327 Fuel Element Graphite (25-g sample)^a

Element	PPM	Element	PPM
Ce	0.1	Sm	<0.02
Pr	0.2	Gd	0.02
Tb	0.2	Dy	0.2
Nd	0.2	La	0.02
Ho	<0.04	Tu	0.02
Lu	0.04	Na	<5.0
Er	0.02	Rb	b
Yb	<0.02	Li	b
Sc	0.02	K	<1.0

^aMethod: spectrographic analysis; accuracy: $\pm 30\%$.

^bNot detected at sensitivity of 2.

Table 7. H-327 Graphite Permeability (Maximum Measured Differences in Helium Flow Constants in Single Log)

Log Position Orientation	End Parallel	End Transverse	Mid-Length Parallel	Mid-Length Transverse
Helium permeability K, cm ² /sec	3.4	1.24	4.2	1.11
F ₀ , latm, cm ³ /sec	2800	1300	1750	1100
Avg. effective pore radius, cm	8.7 x 10 ⁻⁴	7.1 x 10 ⁻⁴	4.2 x 10 ⁻⁴	6.8 x 10 ⁻⁴
Effective No. of pores, h	1.1 x 10 ³	1.05 x 10 ⁵	1.7 x 10 ⁶	1.10 x 10 ⁵
Effective No. of pores per cm ²	2.2 x 10 ³	2.1 x 10 ³	3.4 x 10 ³	2.1 x 10 ³

Table 8. Maximum Measured Differences in Helium Flow Constants Between Logs

Log Position Orientation	End of Log		Mid-Length of Log	
	Parallel	Transverse	Parallel	Transverse
Helium permeability K, cm ² /sec	0.28-4.9	0.66-2.1	0.72-4.2	2.7-3.6
F ₀ , latm, cm ³ /sec	620-2900	420-2300	390-1750	1100-2600
Avg. effective pore radius, cm	3.3-8.7 x 10 ⁻⁴	2.4-7.1 x 10 ⁻⁴	2.8-13.0 x 10 ⁻⁴	4-10 x 10 ⁻⁴
Effective No. of pores per cm ²	0.9-2.2 x 10 ⁴	0.5-2.1 x 10 ⁴	5.8-36.0 x 10 ⁴	1-40 x 10 ⁴

Table 9. Mechanical Strength of H-327 Graphite

	Parallel Orientation		Transverse Orientation	
	Preirradiation	Postirradiation	Preirradiation	Postirradiation
UTS, psi	1629 - 2394	3670 - 4200	924 - 1408	1500 - 2600
E, x 10 ⁶ psi ^a	1.51 - 1.74	2.08 - 3.1	0.58 - 0.68	1.25 - 1.79
Strain at fracture, in./in. x 10 ⁻³	1.4 - 2.1	1.8	2.1 - 3.0	3.2
E secant, x 10 ⁶ psi	1.0 - 1.3	2.0 - 2.3	0.46 - 0.5	0.47 - 0.8

^aChord modulus between 250 and 500 psi.

Fig. 3. Distribution of Tensile Strength at Room Temperature for Production-Grade H-327 Graphite (Parallel to Extrusion in Mid Cross Section of Log).

Fig. 4. Resistivity vs Temperature of Graphitization for Fort St. Vrain Fuel Element Graphite; 18-in. Diameter, Graphitized in Induction Coil Furnace.

Fig. 5. Typical Differences in Uniaxial Tensile Properties at Room Temperature Within H-327 Graphite Log.

Fig. 6. Fort St. Vrain Flux-Temperature Profile for H-327 Fuel Element Material.

Fig. 7. Dimensional Changes in Pilot and Production Grade H-327 Graphite Irradiated at 600 to 700°C. Pilot-scale data from ref. 2.

Fig. 8. Dimensional Changes in Pilot and Production Grade H-327 and Gilsonite Graphites Irradiated at 700 to 800°C. Pilot-scale and Gilsonite data from ref. 2.

Fig. 9. Dimensional Changes in Pilot and Production Grade H-327 and Gilsonite Graphites Irradiated at 800 to 1015°C. Pilot-scale and Gilsonite data from ref. 2.

Fig. 10. Dimensional Changes in Pilot and Production Grade H-327 and Gilsonite Graphites Irradiated at 950 to 1150°C. Pilot-scale and Gilsonite data from ref. 2.

Fig. 11. Uniaxial Tensile Stress-Strain Plots for Irradiated and Unirradiated H-327 Graphite.

Fig. 12. Irradiation Effects on Thermal Conductivity of H-327 Graphite in the Direction Perpendicular to Extrusion.

CHARACTERIZATION OF SEVERAL TYPICAL POLYGRAPHITES
WITH SOME CONVERGENCE ON SOLID BILLET ATJ-S

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ABSTRACT

Several typical fine, medium, and relatively coarse grain graphites are characterized in terms of thermal and mechanical properties, variations in property, correlations of these properties with porosities and other monitors, and their typical flaws and inspectability. These graphites include nominal AXM-5Q, AXF-5Q, ATJ, G90, and solid billet ATJ-S.

The need to characterize the material for a specific application is emphasized. Essentially, the argument is that proper characterization involves, first, the determination of the general property levels for a generic type, then a more intensive look at the critical properties involved in an application, and finally a screening of the uniformity and reproducibility that can be expected. A record of macrostructure and of inspectability by nondestructive test methods must be kept for each specimen, with the record of its properties. Such records enable judgments to be made on the properties of subsequently received large pieces that cannot be destructively tested and from which specimens for extensive testing cannot be cut. Judgments on the properties of such large pieces can be made by comparison of their macrostructure, the results of nondestructive tests, and a few property tests on specimens from selected locations where property "profiles" are known.

The data and observations also should provide interest for those people who are designing hardware or parameter studies for biaxial tests, temperature stress tests, fracture mechanics tests, or impulse loading.

PROPERTIES IN GENERAL

Figure 1 shows a family of stress-strain curves for various graphites at 2000°F to 2500°F. Generally the stiffness, yield, and ultimate strength increase with decreasing maximum grain size for either the with or across grain direction.

Figure 2 shows the 3500°F envelopes, which is where most of the polygraphites seem to obtain maximum stiffness though not necessarily maximum yield or ultimate. ATJ-S becomes a little stiffer than the AXF-5Q or AXM-5Q, and G90 is gaining on the others in terms of strain at failure.

At 4500°F, the envelopes as shown in Figure 3 have changed character considerably. AXF-5Q has extensive "plastic" behavior; ATJ-S has lost a little stiffness relative to AXF-5Q; ATJ has gained in strain at failure in relation to ATJ-S; and G90 has now fallen relatively well behind these other materials in strain at failure. Yield seems to depart from a rather common "elastic" envelope.

Some of the properties of interest to the designer are shown as a function of temperature in Figure 4. Fracture strength at this strain rate is a fairly uniform function of temperature, increasing continuously to 5000°F.

The 0.02 percent yield of these graphites is about 40 percent of ultimate, and again observe the same order, although there is a temperature peak at about 3500°F.

Strain at failure for three graphites follows a fairly uniform pattern, decreasing at first with increasing temperature and then increasing drastically at a strain threshold temperature. The threshold saddle is moving progressively to the left toward a lower temperature for the finer grained graphites. The elastic moduli

for these three graphites are of the same order though in general smaller for the coarser graphites where one suspects more internal accommodation.

Poisson's ratio does provide a nice spread in values with those for AXF-5Q being quite high and those for G90 quite low. In biaxial and in temperature stress tests, Poisson's ratio may be a good parameter to help design tests to study constitutive relations. Recall that graphites are typically transversely isotropic so that, in fact, there are three values of Poisson's ratio and that they are different.

Figure 5 provides a plot of the wide range of values for thermal conductivity that one can obtain for different graphites. The major point here would be that at the lower temperatures, one can have a rather large range of values for conductivity. One can get the impression that this is indeed a parameter that one can control or select; but at the higher temperatures, or the temperatures that control many designs, the range is reduced. AXM-5Q almost uniquely falls outside of the envelope suggesting relatively less graphitization as is indicated by some of its other properties. The thermal conductivities of three typical graphites often used for parameter tests are shown in Figure 6. Some flexibility is provided by the quite lower values for AXF-5Q at the lower temperatures. For a given graphite, density does correlate with thermal conductivity at least statistically. There is not a one on one correlation between specimens from different billets.

There is a rather broad range of values of thermal expansion available as shown in Figure 7 where the value for AXF-5Q is quite high, and the value for the "original" G90 was quite low. The original G90 logs had quite low expansions that have not been

duplicated yet to the author's knowledge. For a given graphite, there does not seem to be a correlation between density variations and thermal expansion.

SPECIFIC PROPERTIES OF SOLID BILLET ATJ-S

Some typical stress-strain curves are shown in Figures 9, 10, and 11 to provide a feel for the scatter typically seen in a given solid billet of ATJ-S. At a given temperature, a given specimen seems to follow the envelope to its failure site rather than systematically be stiffer, more compliant, or otherwise depart from the envelope. Secant modulus at fracture stays somewhat close to unity, but there is a trend for a given scatter in strength to provide a larger scatter in strain at failure. A composite plot of the averages of the stress-strain curves is shown in Figure 12 with similar curves for ATJ. In the composite, it is striking that ATJ-S is quite stiffer than ATJ at the higher temperatures. At above 4500°F, the stiffnesses of both graphites are quite dependent on stress rates over the range of 10 to 500 psi per second. Below 4000°F, there is not much effect.

The compressive behaviors of ATJ-S at different temperatures as shown in Figures 13 and 14 for with and across grain are important in many applications, particularly where a higher stiffness can convert the thermal expansion into higher tensile strains. In compression, as tension, the material gets stiffer up to about 3500°F or 4000°F, and then becomes more compliant and dependent on stress rate.

The 0.02 percent yield in compression is, perhaps, two times the value in tension, and of course the strains at failure, even at room temperature, are an order of magnitude higher.

One can obtain a reasonably good shear modulus from a torsion test although the fracture value probably is no more than a minimum value. Fracture inevitably is a tensile mode. It is quite difficult in the laboratory to apply shear on a specimen without putting an equal or higher tension some place, and the fracture will be at tensile mode. In spite of these limitations, such torsion curves as those shown in Figures 15 and 16 do have an engineering value.

VARIABILITY IN SOLID BILLET ATJ-S

Now consider variability and the problems it brings to the tester, designer, and experimenter in parameter studies. Figure 17 shows the range of average strengths for 20 billets as reported by four different testers in a recent compilation, the range of average strengths for eight typical billets from one tester, and the range of the individual values for each one of seven individual billets from the latter tester. It is apparent that designing to a 99 percent confidence level reduces the design allowable down to below 3200 psi if one cannot truncate bad testing and bad billets.

Figure 18 shows the density and strength profiles that occur within a billet; these are somewhat worse or more systematic than typical. In the upper part of this billet, there is one density and strength profile across the billet on a radius. Lower in the billet, there is another density and strength profile; and at still another level (axial position) of the billet, there is another density and strength profile.

Certainly there are reasons traceable to the original warm pressing as to why these profiles should vary down the billets. There is nothing strange at all about that. The question is whether or not these variabilities are reproducible and the result of

macrostructure. Figure 19 shows typical macrostructures at 100X from various locations in a solid billet indicating high porosity zones that do match the profiles. The property variations, particularly the strength and strain at failure, do relate to these macrostructures as indicated by other extensive work by the author.

Figure 20 is a plot of open porosity (1 to 6 mils in size for ATJ-S) versus strength for some 200 data points and provides a quantitative measure of what one sees under the microscope. Larger open porosity reduces strength. One standard deviation for all of the data truncates every specimen that upon subsequent inspection either had a disparate flaw or high open porosity that would fall within 95 percent confidence limits on the regression of strength versus open porosity. This is a powerful observation if inevitably true, since it means that better inspection of disparates plus a tighter control or inspection of open porosity would permit raising the design allowable from the 3200 psi mentioned earlier to 4200 psi with no loss in confidence.

There are disparate flaws in graphite larger than the larger voids (background voids) associated with the normal porosity. A radiograph of a disparate void is shown in Figure 21. Pulse-echo would not permit the preselection of this disparate as shown in Figure 22 because of the noise or other signals.

In the fracture face of the broken tensile specimen, this disparate is seen in Figure 23 as a void cluster covering perhaps 12 x 40 mils. Its influence on strength is seen in Table 1 where the value for this specimen was 20 percent low for strength and about 40 percent low for strain at failure. Remember that differences seen in strength are magnified in strain at failure because of the nature of the stress-strain curve. The electrical resistiv-

ity of the gage length of the disparate specimen was about the same as that for the average, as was the velocity, so it was not an odd material other than for the disparate void.

CONCLUSIONS

Graphites are continuum in their bulk behavior and have engineering properties. They also have nominal properties for given ranges of grain size. Different graphites have different characteristic flaws and inspectability.

For a given graphite it is necessary to recognize and properly handle both uniformity and reproducibility, particularly if a small factor of safety is required; this is possible by the judicious profiling of properties and monitors coupled with NDT inspection. Both the typical properties and the variability are observed as differences in the macrostructure.

As a final observation, the designer should never use handbook or typical values as given here to design hardware where low factors of safety are involved. This type of design process demands close iteration between the producer, the characterizer, and the systems group conducting the design, particularly in terms of applicable constitutive relations, reproducibility, and judgments on profiles and truncations.

TABLE 1
INFLUENCE OF DISPARATE VOID IN SPECIMEN A, ATJ-S

	Temp	Density	Strength	Modulus	Strain	Electrical Resistivity	Velocity
Average	4000°F	1.827	7460	1.70	7.6	738	0.1049
Spec A	4000°F	1.823	6040	1.80	4.6	740	0.1045

Fig. 1. Typical Stress-Strain Curves for Several Polygraphites.

Fig. 2. Typical Tensile Stress-Strain Curves for Various Graphites.

Fig. 3. Typical Tensile Stress-Strain Curves for Various Graphites With Grain.

Fig. 4. Mechanical Properties of Various Types of Graphites.

Fig. 5. Thermal Conductivity of Various Graphites.

Fig. 6. Thermal Conductivity of Selected Graphites.

Fig. 7. Thermal Expansion of Selected Graphites.

Fig. 8. Tensile Stress-Strain of ATJ-S at 70°F.

Fig. 9. Tensile Stress-Strain of ATJ-S at 1500°F.

Fig. 10. Tensile Stress-Strain of ATJ-S at 2500°F.

Fig. 11. Tensile Stress-Strain of ATJ-S at 3500°F.

Fig. 12. Comparison of Tensile Stress/Strain, With Grain, at Various Temperatures, of ATJ and ATJ-S.

Fig. 13. Compressive Stress/Axial Strain Curves for ATJ-S, Across Grain.

Fig. 14. Compressive Stress/Axial Strain Curves for ATJ-S, With Grain.

Fig. 15. Torsion of ATJ-S, With Grain.

Fig. 16. Torsion of ATJ-S, Across Grain.

Fig. 17. Ranges of Fracture Strengths for ATJ-S Specimens With Grain at 70°F.

Fig. 18. Comparison of Strength and Density Profiles, Radially and Axially, for ATJ-S Billet L-4-6 (With Grain).

Fig. 19. Macrostructure of Billet ATJ-S. 100X. Reduced 5%.

Fig. 20. Strength as a Function of Open Porosity for ATJ-S, With Grain.

Fig. 21. Radiographic Results (0° and 90° Orientations) for Specimen A, Showing Single-Void Strength Reducer.

Fig. 22. Pulse-Echo Smears and Standing Signal for Specimen A.

Short End

Long End

Fig. 23. 10X Photographs of 0.012-inch $\times$ 0.040-inch Void in Specimen A.

MICROSTRUCTURAL ANALYSIS TO DETERMINE ELASTIC MATERIAL
PROPERTIES OF A FILAMENT WOUND GRAPHITE COMPOSITE
HAVING ORTHOTROPIC CONSTITUENTS*

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ABSTRACT

A microstructural analysis was made of a filamentary composite which consisted of a transversely isotropic fiber and a cylindrically transversely isotropic matrix. The properties of the matrix were varied from isotropic to anisotropic conditions while holding the fiber properties constant. Then the matrix properties were held constant, and different fibers were used. Longitudinal and transverse direction composite moduli and the associated Poisson's ratios were calculated. The stress distribution throughout the composite due to transverse loading and a stress concentration factor were calculated. A two-dimensional finite element computer program was used to solve the problem.

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Introduction

The microstructure of a composite composed of graphite fibers and a chemical vapor deposited (CVD) carbon matrix was analyzed to obtain macroscopic material properties of the composite. Five different fibers were used in the analysis: CY2-5, Thornel 25, Thornel 50, Morganite Type I and Morganite Type II. In consideration of the nature of the CVD process and from observations of photomicrographs of filament wound CVD carbon composites, the matrix appears to be cylindrically transversely isotropic, i.e., the matrix has the same properties in the axial and circumferential directions but a different set of properties in the direction normal or radial to the fiber. The fibers are believed to be transversely isotropic, with the plane of isotropy perpendicular to the fiber axis.

Whitney's work (Reference 1) was apparently the first micromechanics investigation which considered a transversely isotropic fiber with an isotropic matrix. Other investigators also included the effect of transversely isotropic fibers in their micromechanics analyses (References 2, 3, 4, 5 and 6 for circular cross-section fibers, Reference 7 for rectangular and Reference 8 for fibers of elliptical, square, and rhombic cross sections). Hermans (Reference 9) considered both a transversely isotropic matrix and fiber. This paper extends micromechanic analysis to the case of a cylindrically transversely isotropic matrix and a transversely isotropic fiber.

The elastic properties of the composite E_L , E_T , ν_{LT} , ν_{TT} , were determined where the L and T directions are defined as in Figure 1. The fibers were assumed to be very long compared to their diameter, regularly spaced as in Figure 1, and circular in cross section. With these assumptions, planes of symmetry were available to reduce the composite to a model which considers only one-fourth of a fiber and its surrounding matrix, as shown in Figure 2. These

planes of symmetry exist when the composite is loaded in the L or T directions. Composite dimensions were determined using a 50% fiber volume. The orientation of the principal directions of the material properties for the matrix and composite is also shown in Figure 2. The r- direction is parallel to the L-axis.

The analysis was conducted in two parts. The first step was to fix the properties of the fiber and vary the properties of the matrix. The properties used were those of a CY2-5 fiber which had been heat-treated at 3000°C for two hours. The properties of all the fibers used in this work were either estimated or determined experimentally (Reference 10). The properties of the matrix were varied from isotropic conditions to an orthotropic case representing pyrolytic graphite. The material properties for the matrix were assumed to be cylindrically transversely isotropic where $E_n = E_r \neq E_s$. The study was made by changing the ratio of E_s/E_n from 1 to 4 with the restriction that $E_s + E_n = 7.5 \times 10^6$ psi. The material properties of the matrix are shown in Table 1. Also, one case (3a) was analyzed with the ratio $E_s/E_n = 3$, $E_s = 3 \times 10^6$ psi and $E_n = 1 \times 10^6$ psi. The Poisson's ratios and shear moduli were linearly adjusted from the values shown for $E_s/E_n = 4$ to those of $E_s/E_n = 1$. The properties of the CY2-5 fiber are shown in Table 2.

The second part of the analysis consisted of fixing the matrix properties and using different fibers. The definition of the matrix properties, Case 3a, was chosen; the properties of the fibers used are shown in Table 2.

SOLUTION TECHNIQUE

The material properties E_L , E_T , ν_{LT} and ν_{TT} were determined by modeling the fiber and matrix with finite elements as shown in Figure 3 and using the SAAS II computer code (Reference 11). A material axis orientation angle, β , was selected for each element. The structure was deformed by a constant strain in one T-direction, T_1 , and the strain in the second T-direction, T_2 , was held constant. A condition of generalized plane strain was imposed in the L-direction, $\epsilon_L = \text{constant}$. The stress-strain relationship takes the form of Equation (1).

$$\begin{bmatrix} \sigma_{T_1} \\ \sigma_{T_2} \\ \sigma_{T_1 T_2} \end{bmatrix} = \begin{bmatrix} c_{11} & c_{12} & c_{14} \\ c_{21} & c_{22} & c_{24} \\ c_{41} & c_{42} & c_{44} \end{bmatrix} \begin{bmatrix} \epsilon_{T_1} \\ \epsilon_{T_2} \\ \epsilon_{T_1 T_2} \end{bmatrix} \quad (1)$$

$$-\Delta T \begin{bmatrix} \psi_1 \\ \psi_2 \\ \psi_4 \end{bmatrix} + \epsilon_L \begin{bmatrix} c_{13} \\ c_{23} \\ c_{43} \end{bmatrix}$$

where c_{ij} and ψ_i are defined for an orthotropic material.

When ϵ_L is assumed to be restrained by a body force, the problem can be formulated as one in plane strain with $\epsilon_L = 0$. The value of ϵ_L is found iteratively and Equation 1 is used to evaluate the stresses; ϵ_L is evaluated at successive iterations with the use of Equation 2.

$$\int_A \sigma_L dA = \int_A (C_{13} \epsilon_{T_1} + C_{23} \epsilon_{T_2} + C_{33} \epsilon_L + C_{43} \epsilon_{T_1 T_2} - \Delta T \psi_3) dA \quad (2)$$

A correct value of ϵ_L occurs when the right-hand side of Equation (2) is equal to zero; ϵ_L is evaluated at the end of each iteration with Equation (3).

$$\epsilon_L = \frac{- \int_A (C_{13} \epsilon_{T_1} + C_{23} \epsilon_{T_2} + C_{43} \epsilon_{T_1 T_2} - \Delta T \psi_3) dA}{\int_A C_{33} dA} \quad (3)$$

The calculated value of ϵ_L is used for the next iteration until Equation (4) is satisfied.

$$\left| \int_A \sigma_L dA \right| \leq X \quad (4)$$

X is the resulting force in L direction, which is made arbitrarily small. When the force in the L-direction is zero, Equations 5a, 5b, and 5c can be written for the composite.

$$\epsilon_{T_1} = \frac{\sigma_{T_1}}{E_T} - \frac{\nu_{TT} \sigma_{T_2}}{E_T} \quad (5a)$$

$$\epsilon_{T_2} = - \frac{\nu_{TT} \sigma_{T_1}}{E_T} + \frac{\sigma_{T_2}}{E_T} \quad (5b)$$

$$\epsilon_L = - \frac{\nu_{TL} \sigma_{T_1}}{E_T} - \frac{\nu_{TL} \sigma_{T_2}}{E_T} \quad (5c)$$

Equations 5a and 5b can be solved simultaneously for E_T and ν_{TT} because ϵ_{T_1} and ϵ_{T_2} are applied strains in the T_1 and T_2 directions and σ_{T_1} and

σ_{T_2} are the average stresses in the T_1 and T_2 directions at the boundaries. Then ν_{TL} can be determined from Equation 5c.

To calculate E_L , a plane strain condition ($\epsilon_L = \text{constant}$) is imposed upon the model. When $\epsilon_L = 0$, and ϵ_{T_1} and ϵ_{T_2} are constants, Equation (6) results.

$$\frac{\sigma_L}{E_L} - \frac{\nu_{TL} \sigma_{T_1}}{E_T} - \frac{\nu_{TL} \sigma_{T_2}}{E_T} = 0 \quad (6)$$

The value of σ_L is determined by Equation (7).

$$\sigma_L = \frac{\sum_{n=1}^m \sigma_{L_n} A_n}{\sum_{n=1}^m A_n} \quad (7)$$

where m is the number of elements.

Equation (6) can be solved for E_L , and then ν_{LT} can be obtained from Equation (8).

$$\nu_{LT} = \frac{\nu_{TL} E_L}{E_T} \quad (8)$$

A stress concentration factor was obtained from Equation (9) for loading of the composite in only one T-direction. A condition of generalized plane strain was imposed on the other T-direction and the L-direction.

$$K = \frac{\sigma_{\text{maximum}}}{\sigma_T} \quad (9)$$

σ_{maximum} is the maximum principal stress in the $T_1 - T_2$ plane, and σ_T is the average stress resulting from a constant strain applied in the T direction.

This analytical technique was verified by comparing a sample calculation with the values obtained elsewhere for a composite (Reference 12). The case of an aluminum fiber and Hysol 4290 epoxy matrix with a fiber volume of 65% was modeled and analyzed. The theoretical results of Reference 12. predict a stress concentration factor of 1.9 for a load in a transverse direction, although the experiment reportedly gives about 1.85. A value of 2.03 was predicted by the finite element method used in this investigation. The distribution of the stress also agreed with the results in Reference 12, as shown in Figure 4.

RESULTS

The results are shown in Table 3 for E_T , E_L , ν_{LT} , ν_{TT} , and K for all the combinations of matrix and fiber properties which were considered.

Figure 5 shows the material properties as a function of E_s/E_n where $E_s + E_n = 7.5 \times 10^6$ psi and the CY2-5 fiber is used. Contour plots of K are given in Figures 6 through 14.

CONCLUSIONS

The material properties predicted with the CY2-5 fiber and $E_s + E_n$ of the matrix equal to 7.5×10^6 psi do not correlate with the values predicted from tests conducted on filament wound cylinders (Reference 13). The values predicted by the tests were $E_L = 4.5 \times 10^6$ psi, $E_T = 2.0 \times 10^6$ psi, and $\nu_{LT} = 0.52$. To predict the experimental values of E_L and E_T , it was necessary to reduce the moduli of the matrix to the values of $E_s = 3.0 \times 10^6$ psi and $E_n = 1.0 \times 10^6$ psi. The value of ν_{LT} was not predicted.

The results also show that the rule of mixtures does not accurately predict the values of the modulus in the L-direction when the matrix is highly anisotropic with the CY2-5 fiber. A value of 5.5×10^6 psi would be predicted for $E_s/E_n = 4$ case by using a rule of mixtures, whereas the finite element obtained value is 5.825×10^6 psi. However, a rule of mixtures result is closely approximated when the matrix is isotropic. This conclusion was also arrived at by Chen and Cheng (Reference 6) for a hexagonal fiber array with an isotropic matrix and a transversely isotropic fiber. A value of $E_L = 4.375 \times 10^6$ psi is obtained by using a rule of mixtures calculation for $E_s/E_n = 1$, as compared to 4.414×10^6 psi for the finite element calculation. The most important trend predicted by the analysis is that both the stress concentration factor, K, and E_L are reduced when the matrix is made less anisotropic.

When the fiber is changed and the matrix held constant, several conclusions can be drawn. First, it is seen that E_T does not vary greatly. Since the transverse strength of the composite will be inversely proportional to the change in the quantity K, a composite made of TH-50 would have 15% less strength in the transverse direction than the composite made of CY2-5 fibers, considering zero residual stresses due to manufacturing processes. Further, E_L can be very accurately predicted with a "rule of mixtures" calculation when high modulus fibers are used. Except for the CY2-5, ν_{LT} of the composite does not change appreciably when fibers are changed.

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Table 1. Matrix Material Properties

$\frac{E_s}{E_{n \text{ matrix}}}$	E_n psi $\times 10^6$	E_s psi $\times 10^6$	E_r psi $\times 10^6$	$\nu_{sn} = \nu_{rn}$	ν_{rs}	G_{ns} psi $\times 10^6$
1	3.75	3.75	3.75	0.2	0.2	1.5625
2	2.5	5.0	5.0	0.45	0.083	1.10833
3	1.875	5.625	5.625	0.7	-0.033	0.65417
4	1.5	6.0	6.0	0.95	-0.15	0.2000
3a	1.0	3.0	3.0	0.7	-0.033	0.5000

Table 2. Fiber Material Properties

Name	E_r psi $\times 10^6$	$E_s = E_n$ psi $\times 10^6$	ν_{sn}	$\nu_{rn} = \nu_{rs}$	G_{ns} psi $\times 10^6$
CY2-5	5.0	3.25	0.2	0.4	1.35
Thornel 25	23	2.3	0.5	0.10	1.2
Thornel 50	48	2.1	0.2	0.08	1.0
Type I	61	3.1	0.2	0.06	0.9
Type II	34	5.6	0.5	0.20	1.4

Table 3. Composite Material Properties

Fiber	$\frac{E_s}{E_{n_{matrix}}}$	E_L psi $\times 10^6$	E_T psi $\times 10^6$	ν_{LT}	ν_{TT}	$K_{max} = \frac{\sigma_{maximum}}{\sigma_T}$
CY2-5	1	4.414	3.496	0.301	0.203	1.116
CY2-5	2	5.145	3.279	0.313	0.299	1.526
CY2-5	3	5.627	2.857	0.318	0.406	1.916
CY2-5	4	5.829	2.135	0.321	0.563	2.858
CY2-5	3a	4.185	2.134	0.339	0.328	1.343
Thornel 25	3a	13.044	2.001	0.199	0.438	1.402
Thornel 50	3a	25.533	1.882	0.182	0.365	1.554
Type I	3a	32.037	2.045	0.179	0.401	1.379
Type II	3a	18.580	2.484	0.225	0.444	1.301

Fig. 1. Fiber-Matrix Arrangement in Composite.

Fig. 2. Sign Conventions and Dimensions of Model.

Fig. 3. Finite Element Mesh.

Fig. 4. Interfiber Stress; Test vs Theory (Fiber Volume = 65%).

Fig. 5. Material Properties E_T , E_L , ν_{LT} and Stress Concentration Factor K_{max} for $E_s + E_n = 7.5 \times 10^6$ psi, CY2-5 Fiber.

Fig. 6. Contour Plot of K for $E_s/E_n = 1$, $E_s + E_n = 7.5 \times 10^6$ psi, CY2-5 Fiber.

Fig. 7. Contour Plot of K for $E_s/E_n = 2.0$, $E_s + E_n = 7.5 \times 10^6$ psi, CY2-5 Fiber.

Fig. 8. Contour Plot of K for $E_s/E_n = 3.0$, $E_s + E_n = 7.5 \times 10^6$ psi, CY2-5 Fiber.

Fig. 9. Contour Plot of K for $E_s/E_n = 4.0$, $E_s + E_n = 7.5 \times 10^6$ psi, CY2-5 Fiber.

Fig. 10. Contour Plot of K for $E_s/E_n = 3.0$, $E_s + E_n = 4.0 \times 10^6$ psi, CY2-5 Fiber.

Fig. 11. Contour Plot of K for $E_s/E_n = 3.0$, $E_s + E_n = 4.0 \times 10^6$ psi, Thornel 25 Fiber.

Fig. 12. Contour Plot of K for $E_s/E_n = 3.0$, $E_s + E_n = 4.0 \times 10^6$ psi, Thornel 50 Fiber.

Fig. 13. Contour Plot of K for $E_s/E_n = 3.0$, $E_s + E_n = 4.0 \times 10^6$ psi, Morganite Type I Fiber.

Fig. 14. Contour Plot of K for $E_s/E_n = 3.0$, $E_s + E_n = 4.0 \times 10^6$ psi, Morganite Type II Fiber.

HOLOGRAPHIC INTERFEROMETRIC STUDIES
OF CARBON COMPOSITE CYLINDERS*

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ABSTRACT

Initial feasibility studies on carbon composite cylinders indicated that the location of failure corresponded to highly deformed areas which were previously located by holographic interferometry at low stress levels. Calibration studies on aluminum cylinders were then done to ascertain the limitations and experimental errors in the testing procedures. Correspondence was obtained between circumferential strain values obtained by interferometry and by strain gages. Perspective angle and spherical wave corrections were necessary to achieve a high degree of accuracy at the viewed extremes of the cylinders. Emulsion shrinkage was found to distort the images on real-time studies.

Anomalous points on the cylinders could be easily found.

INTRODUCTION

Preliminary investigations on the application of laser interferometry to the inspection of carbon-carbon composites indicated that anomalous areas of a hollow cylinder could be found. An internal pressure via a bladder arrangement was used to load the cylinders in a nondestructive manner. Later destructive evaluations indicated that fracture occurred at an anomalous point determined previously by the low-stress loading.

The anomalous areas of the cylinders were determined by calculating the circumferential strain variations along circumferential belts of the cylinder. Methods of geometric optics were used to calculate the variation in the radius of the cylinders on selected

* This work was supported by the U. S. Atomic Energy Commission.

circumferential belts. A standard expression was then used to relate circumferential strain to the radius variation. Several assumptions were made to simplify the analysis and the effects of the simplifications were evident from discrepancies in the strain values found at identical points from overlapping holographic views. A study was then undertaken to evaluate the effects of the various simplification assumptions. Also, it was determined necessary to evaluate the accuracy of measurement of absolute versus relative deformations of the cylinder.

PRELIMINARY INVESTIGATIONS

A carbon-carbon test cylinder* was inspected by holographic interferometry at 90° intervals of rotation. The cylinder was deformed by a bladder arrangement with an internal pressure change of 40 psi. A cross-section of the test cylinder and bladder arrangement is shown in Fig. 1. The circumferential strain variations about some average value were calculated from the fringe patterns. Two overlapping views both indicated either an excessive strain or a minimum strain in the overlapping region. Although both views indicated the anomalous strain of the same sense, the numerical values differed. The strain values were relative circumferential strain about a center belt. The simplifying assumptions made in the analysis evidently destroyed accuracy at the extremes of the viewed areas of the cylinder. By extending the curve connecting the calculated strain values from each overlapping view until they intersect, the curve of Fig. 2 results. Note that the relative position of the minimum of the strain curve and the fracture location coincide. The fracture position on the cylinder was determined by a later destructive test. If the opposite sense of deformation were assumed, the curve of Fig. 2 would be inverted predicting a maximum strain at the point of fracture. The actual sense of relative surface motion was not known since only one view of the hologram was available.

Other shaped samples were also inspected holographically and then tested to destruction. A more ductile material was used in these samples. The general shape was a hollow cylinder with one closed end. A distributed axial loading was used for both nondestructive and destructive testing. The material was carbon cloth with an epoxy matrix. Figure 3 shows the relative circumferential strain along a circumferential belt near the failure belt. The center of the fracture is noted on the figure and again this corresponds with the location of a strain anomaly.

FACTORS AFFECTING THE ACCURACY OF THE ANALYSIS

In analyzing many axi-symmetric test samples nondestructively and then testing destructively, several possible areas of inaccuracy were noted. These subject areas are as follows:

1. Perspective angle existed in viewing the hologram and affects the calculation of the deformation.

* Holograms were made by Battelle Memorial Institute, Columbus, Ohio.

2. Spherical wave effects resulting from illuminating an object through a spacial filter versus a collimator affects the deformation calculations.
3. Any sample translation and/or rotation creates extra fringes and confuses deformation calculations.
4. Lateral deformation from Poisson's ratio effects distort the fringe patterns and affect relative strain values between circumferential belts.
5. Partial fringe estimations must be made in some instances and this affects accuracy of calculations.

The first two problem areas itemized are mathematically identical when one accounts for them in the deformation analysis. The perspective angle problem relates to the paths taken by reflected light from the object. These paths are not parallel since the camera or eye viewing the object through the hologram looks through a small window or area of the hologram. This fact is schematically illustrated in Fig. 4. The iris of the camera cannot be too large or a Fresnel zone is exceeded in size and the coherence will be insufficient to see the fringes. This means that a camera placed near the hologram plate cannot have its f-stop iris opened beyond a limiting diameter or the fringes cannot be seen in the photograph although the eye can view them with its small iris opening. The perspective angle effects are geometrically similar to the spherical wave illuminating problem as shown by Fig. 5.

In deriving the geometric relationships relating the fringe pattern to deformation of the cylinder surface, the following simplification assumptions were made.*

1. Illumination and viewed wave fronts were considered to be planar.
2. Axial displacement was neglected.
3. Deformation was considered radial.
4. A zero order fringe was assumed and used as a reference.

These assumptions allowed one to calculate surface radial deformation on circumferential belts of the cylinder with only one measurement taken from a photograph of the fringe pattern. Variation in deformations along the axis of the cylinder was determined by analyzing many circumferential belts spaced along the length of the cylinder. Relative deformation and strain values were thus available but no absolute values could be calculated unless one knew the exact fringe order.

In extending the deformation analysis to cones of small half-angle, axial deformations resulting from internal bladder loading along with cumulative axial displacement from Poisson's ratio effects greatly affect the fringe pattern and thus the accuracy of the calculated deformations. It should be noted that any rotation and/or translation of the test object greatly affects the fringe

* Derivation shown in SC-DR-69-281, "Holographic Inspection of the Deformation Patterns of Carbon-Carbon Cylinders Under Load," O. J. Burchett and J. L. Irwin, April, 1970.

patterns. Thus fixturing to hold the sample from unwanted movement during loading is paramount.

If one wishes to determine the deformation pattern resulting from a very slight change in loading, only a few fringe orders exist on the interference hologram. One must then estimate the fringe order between bright and dark fringes at desired points. Some error must accompany this action. It should also be noted that the data taken from the pictures of the hologram requires a position measurement which relates the fringe to some reference. One must estimate the center of the fringe and errors always result. Also, the fewer the number of fringes the broader the fringes and the more difficult it is to locate the maximum extinction point.

Any loading bladder, point load, line load, etc., used to deform the part must necessarily have an effect on the deformation of the part. A bladder may reduce the internal pressure applied to the actual part or perturbate the pressure thus affecting both relative and absolute deformation or strain values. For a given sample shape and loading configuration, one must determine these error effects in order to increase interpretation accuracy.

DETERMINATION OF BLADDER AND EDGE EFFECTS FOR A CARBON-CARBON CYLINDER

The desired end result was to ascertain the quality of a carbon-carbon test cylinder without damage. The sample was to be loaded with an internal bladder since it was to be tested for mechanical properties later with the same bladder. It was also desired to ascertain if the bladder affected the loading applied to the sample, and to ascertain if anomalous areas of the cylinder could be correlated with later failure areas.

Not all of the above mentioned goals could be accurately determined with a limited number of test samples; therefore, the first step was to evaluate the limits of test accuracy related to the bladder effects and to the holographic strain analysis. It should be noted that several previous relationships between anomalous points and later failure at that area had been established. The unknown factors in those tests were the accuracy of deformation analysis and too few test samples for positive conclusions.

The effects of the loading bladder were to be determined by first testing an aluminum test cylinder (well characterized material) both with the bladder loading and then with an O-ring seal technique. Thus, the cylinder would be loaded both with the bladder and without the bladder. Comparisons of the circumferential strain readings with identical internal pressures would indicate any bladder effects. Also, the carefully machined aluminum cylinder should have constant deformation and circumferential strain around belts. If the holographic strain analysis did not correspond on the same circumferential belts, then corrections to the analysis method would be required. The same test procedures should then be applied to the carbon-carbon cylinder for evaluation.

RESULTS AND CONCLUSIONS

The test results from the aluminum cylinder indicated that the circumferential strain was within the ± 2 count accuracy of the strain gage reader for the "bladder" or "no bladder" conditions. Table 1 tabulates both the strain gage readings and resulting differences. The results of the tests on the aluminum cylinder indicate that the effects of the bladder in the resulting strain (circumferential) can be neglected.

Figure 6 shows the comparison of circumferential strain values calculated from holographic interferometry, p_r/Et , and those read from the strain gages all on the strain gage belt. Figure 7 shows that the strains compare within one or two μ -in/in. when the strains are calculated at the maximum fringe order and then compared with the strain gage readings. Double exposure holograms accurately determine the circumferential strain over the major portion of the illuminated area when applying spherical wave and perspective angle corrections. Real-time holograms have the advantage of allowing one to accurately determine the fringe order and to determine the direction of surface undulation. In fact, the correct order for the carbon-carbon cylinder fringe pattern was determined from a real-time viewing of the fringe system. However, the real-time holograms consistently predict strain values accurately only at the point of maximum fringe order. Emulsion shrinkage effects are the main cause of error in that the reference image is distorted and the focal plane of the fringes does not correspond with the plane of the object.

Calibration tests on the carbon-carbon cylinder indicated that the bladder had little or no effect on strains. The porous material along with surface pits at the winding pattern cross-overs prevented a tight fluid seal without some axial constraint of the cylinder. The conclusion resulting from the tests was that the bladder was the best method of loading the cylinder. Figure 8 shows the values of the strains along a circumferential belt passing through three strain gages. The average of the three strain gage readings are also plotted on Fig. 8 as a band which includes the ± 2 count accuracy of the strain gage reader. The two strain measuring methods correspond satisfactorily over about 70° of the viewed angle of the cylinder.

Table 1. Aluminum Test Cylinder

Pressure Seal Method	Internal Pressure psig	Circumferential Strain μ -in/in				Avg. Circ. Strain	Combined Test Avg.	Bladder Effect μ -in/in	Percent Bladder Effect
Bladder	398.4	456	470	448	458	459	459	3.56	0.77
Bladder	398.4	458	472	448	459				
Bladder	399.2	456	472	448	459				
O-Ring	399.6	458	474	450	461				
O-Ring	399.4	460	474	454	463				
O-Ring	399.8	462	472	454	463				
Bladder	199.4	225	235	223	228	228	228	1.17	0.51
Bladder									
O-Ring	199.9	227	236	225	229				
O-Ring	199.0	226	235	224	228				
Bladder	100.0	111	116	109	112	112	112	0.50	0.44
Bladder	99.3	111	115	109	112				
Bladder	100.1	111	116	110	112				
O-Ring	99.9	111	117	110	113				
O-Ring	99.7	110	116	111	114				
Bladder	50.1	54	57	53	55	54	54	1.33	2.40
Bladder	50.2	54	57	51	54				
O-Ring	49.9	55	57	54	55				
O-Ring	50.1	55	58	55	56

Fig. 1. Test Cylinder and Bladder.

Fig. 2. Circular Strain of Test Cylinder.

Fig. 3. Circular Strain of Closed-Ended Cylinder.

Fig. 4. Perspective Angle Geometry.

Fig. 5. Similarity of Spherical Wave and Perspective Angle Effects.

Fig. 6. Holographic Strains at Strain Gage Line.

Fig. 7. Holographic Strains at Maximum Fringe Order.

Fig. 8. Carbon Cylinder Circumferential Strain.

✓ ULTRASONIC CHARACTERIZATION AND COMPUTERIZED
ANALYSIS OF GRAPHITE BILLETS* 7382-0

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ABSTRACT

A computer aided, ultrasonic test system for graphite billets has been developed, the primary purpose of which is to select the optimum location within the billet of a re-entry vehicle nose tip. An on-line mini-computer provides process control functions and initial data processing. Further data processing and billet quality analysis are provided by a large computer. Visualization of the internal structure is aided by the use of a computer-controlled plotter.

The ultrasonic test system consists of a commercial ultrasonic tester with an automatic scanning and immersion system. Position readout indicators provide scan position and part rotation information. A special interface module receives ultrasonic test data from the tester and converts this information to analog voltages, while an auxiliary function module, controlled by the mini-computer, digitizes and records the position and ultrasonic information on magnetic tape.

The large computer analyzes the ultrasonic flaw data in terms of the up to 3300 possible nose-tip locations within the billet. In evaluating each particular nose-tip location, every flaw indication is assigned a weight. These weights are totaled for that cone location. The 15 positions having the lowest weights are presented to the design engineer for his final selection.

The plotter is used to provide graphical pictures of the flaws within the billet to aid the engineer in his selection of the optimum position of the nose tip. Plots are made showing all of the flaws in the billet by subdividing into slabs which run parallel to the billet axis and into discs perpendicular to the axis. Such plots show the flaw distribution in a clear, concise manner. Further programs plot histograms of the data with respect to three location parameters.

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INTRODUCTION

Reentry vehicles and various other vehicle applications require nose tips which can survive the high thermal and mechanical stress levels encountered during the reentry environment. Failure of the nose tip and/or its associated heat shield would normally result in the loss of the vehicle or in a severely altered mission. Therefore, the integrity of the nose tip must be maintained.

Graphite has proved to be a useful material in nose-tip applications; however, completely homogeneous graphite is very difficult to acquire. ATJ-S graphite, manufactured by the Union Carbide Company using proprietary processes,³ was selected for our application. Other types of graphite were also considered for the reentry vehicle application.

Graphite strength properties are drastically altered by discontinuities in the material. These discontinuities, which are prevalent in the material, may be noncarbon inclusions, high-density carbon inclusions, voids, porous areas, cracks, low-density areas, or sudden density changes. More broadly, any variation in the material which could be a stress riser or contribute to a loss of cross-sectional area could be detrimental to the end use.

Recognizing that a billet of ATJ-S (Fig. 1) contains many discontinuities, it is then desirable to locate these discontinuities and to position the nose tip in such a manner as to avoid including any discontinuity in the finished part. Should this be unfeasible, it is desirable to avoid clustered discontinuities and to determine the location of those flaws remaining in the nose tip.

Because of a shortage of time for our test program and an even greater shortage of material, no definitive attempt was made to correlate ultrasonic echo characteristics to flaw characteristics. A report by Southern Research Institute⁴ indicates that ultrasonic echoes are indicative of true discontinuities in the material. The approach taken at Sandia was that any discontinuity was undesirable in our nose-tip application.

An ultrasonic nondestructive testing system was developed to locate all such discontinuities, record certain characteristics, and analyze these by use of a computer. The primary test system consists of fixturing, an ultrasonic test system, a signal processing interface, an on-line computer, and peripheral equipment. In addition to the primary test system, a large computer and computer-plotter are used for analysis.

PURPOSE OF THE REPORT

The purpose of this report is to describe the nondestructive testing and data analysis system used for evaluating the quality of ATJ-S graphite billets and to discuss the test results. The system will be described in two sections.

The primary test section will include only the on-line test equipment, and the final data analysis section will describe the programming for the full-size computer and the computer-plotter.

PRIMARY TEST SYSTEM

Our primary test system as shown in Fig. 2 consists of an ultrasonic test system, an electrical interface, an on-line computer, an input-output console, and a rack of peripheral equipment including a magnetic tape recorder.

Ultrasonic Test System

The ultrasonic test system consists of an immersion tank with associated fixturing and the ultrasonic tester. The immersion tank (Fig. 3) provides a mechanical system for both the test item and ultrasonic probe manipulation. The actual water tank is 3 feet deep, 4 feet wide, and 6 feet long. Water, immersing the test billet, provides coupling of the sound wave from the ultrasonic transducer into the billet. Absorption of water by the graphite billet is prevented by a strippable coating of polyvinyl chloride.

A billet is placed on a set of rollers (Fig. 4) which can incrementally or continuously rotate the part, while a potentiometer provides electrical analog signals of the rotational position of the billet. Angular increments are determined at the control panel on the bridge.

A traversing bridge spans the tank and, ordinarily, is free to roll to and fro. However, for this test, it is fixed to locate the transducer over the billet. The transducer is mounted on a search tube held in a manipulator (Fig. 5) which is, in turn, mounted on the bridge. The search tube can be moved back and forth axially, over the part, and a potentiometer provides an analog indication of the distance of the transducer from the end of the billet.

In the scanning operation, the ultrasonic transducer is positioned over the billet and perpendicular to it. The transducer moves, automatically, from one end of the billet to the other, stops, and retraces the path. During the pause at the end of the billet, the billet is rotationally incremented. On the retrace, then, the sound beam is scanning new material.

Bridge controls (Fig. 6) provide variable angle incrementing, variable transducer scanning speed, and selective start and stop positions. Manual controls on the search tube manipulator provide variable transducer-to-billet spacing (water travel) and variable entry angle.

An Immerscope* 424D ultrasonic tester (Fig. 7) is used to obtain the defect indications. A high-voltage pulse is fed to a piezoelectric crystal in the ultrasonic transducer located in the immersion tank. The crystal vibrates at its resonant frequency for a few cycles creating a pulse of sound in the water. The crystal used in this application is a focused, 5-MHz, lithium sulfate disc with a 4-inch focal length in water. The focal spot size is 0.10 inch.

Sound pulses travel through the water and impinge on the graphite billet. A portion of the sound penetrates the billet while a large portion is reflected. Ratio (R) of reflected-to-transmitted amplitudes is determined by the acoustic

*Commercial names throughout this report are used for identification purposes only and their use does not necessarily constitute an endorsement.

impedance (Z) mismatch. In water, where ρ is the density (gm/cc) and V is the sound velocity (in/sec),

$$Z_1 = \rho V = 1 \times 60,000 = 60,000 \text{ gm-in/cc-sec.}$$

In the billet, nominally,

$$Z_2 = \rho V = 1.8 \times 90,000 = 162,000 \text{ gm-in/cc-sec.}$$

Then,

$$R = \frac{Z_2 - Z_1}{Z_2 + Z_1} = \frac{162,000 - 60,000}{222,000} = 0.46.$$

Therefore, 46 percent of the incident wave amplitude is reflected and unusable for flaw detection.

Of the remaining 54 percent, a small portion may be reflected by a flaw. In addition to the interface losses, the signals are further attenuated by the graphite material. That portion of the echo which does return to the transducer creates a slight mechanical stress on the crystal which causes a low amplitude voltage to be generated. The Immerscope takes the echo-generated voltage signals from the transducer, amplifies, rectifies, and integrates them and displays them on the cathode ray tube (CRT). Instrumentation controls are set such that the first echo appearing on the tube is from the front surface with the flaw echoes appearing later in time. Time delay between the front surface echo and the flaw echoes is directly proportional to the depth of the flaw from the outside surface. The constant of proportionality is the sound velocity in the part.

It is interesting to note that, when evaluating graphite materials, an abundance of "noise" echoes is present. These signals originate from minute interfaces in the material and are of interest. However, because of the tremendous number of these, some are disregarded as explained in the section on the interface circuits.

The transducer is pulsed 200 times per second with each pulse lasting 2 to 3 microseconds. The remainder of the time, about 5000 microseconds, it is quiescent and waiting for echoes. As these echoes are received and displayed on the CRT, they are also transmitted, in parallel, to the interface for further processing.

Interface System

The interface system (Fig. 8) provides the mating function between the ultrasonic tester and the computer. Three main functions are:

- . Signal amplitude discrimination.
- . Signal amplitude and location analog conversions.
- . Computer control-signal generation.

Signal amplitude discrimination determines whether a particular ultrasonic signal will be considered. All signals with amplitude less than a predetermined level are rejected, and this level is determined by the echo amplitude from ultrasonic standard test blocks. For example, assume that the minimum echo

acceptance level will be equal to the echo amplitude from a flat-bottom hole 0.030 inch in diameter. If the amplitude of the echo from such a standard hole is 40 percent of full scale on the CRT, the accept-reject level in the interface is set at 40 percent. Signals greater than this are processed; lesser signals are squelched.

In the amplitude and location conversion system, the time from the front surface echo to the flaw echo is converted to a proportional DC voltage. Since the velocity in the material is measured prior to this test, the DC voltage is proportional to the depth of the flaw. A second conversion equating the amplitude of the echo to a DC voltage is also made.

The interface (Fig. 9) operates as follows. The first signal or front surface echo is fed by the splitter to a ramp generator where a voltage ramp is started and fed to the depth peak-readers which begin reading the ramp. The second signal or first flaw echo is fed to a depth peak-reader at which time the ramp is inhibited to that reader and the voltage level at that time is held. The third signal or second flaw echo is fed to the second depth peak-reader and the operation is repeated.

Five channels are available to read five flaw echoes occurring from the same sound pulse. (During manual scanning operations previous to the construction of this system, multiple flaws had been noted with the transducer fixed in position.)

Simultaneously, with the peak-reading operation, the echoes are fed to the amplitude peak-readers where the peak amplitude is read and held. As echo signals are received, a signal is sent to the computer by the computer input control, informing the computer that test data are available and which data channels to interrogate.

Computer and Recording System

Our on-line computer (Fig. 10) is a Honeywell H-21 having an 18-bit word, 4096-word memory bit, 6-microsecond cycle time. Sixteen priority interrupt channels are available. An ASR 35 teletype provides input/output communication with the computer.

Other peripheral equipment includes a 40-microsecond converter with 16 A/D channels and seven D/A channels. A 100-step per second, incremental drive, digital, magnetic-tape recorder is also provided.

This system provides several functions:

- . System control.
- . Analog-to-digital data conversion.
- . Data formation.
- . Data recording.
- . Data real-time print-out.

Data recorded by the system consists of the billet description and the flaw descriptions. The billet is described by its radius and length, while each flaw is described by four parameters. Angular position and distance from the end as determined by the fixture potentiometers, and depth and amplitude as determined by the ultrasonic tester and the interface serve to describe the flaws.

Operation of the computer is as follows. At the "run" signal, the computer commands the bridge to scan the billet. Data from the interface and the position indicators are stored during the scan. As the transducer reaches the end of the billet, an end-of-part (EOP) signal is generated, stopping the scan. The computer examines all the data, discriminates against some, organizes the data into the proper format, and records it. At the option of the operator, a real-time "quick look" print-out on the teletype may be performed in either octal or decimal numbers or both. At the end of the test, the operation is stopped and the operator is informed, via the teletype, of the number of flaws recorded and the number of sweeps in the 360-degree rotation.

The discrimination mentioned earlier rejects certain undesired signals. Signals occurring during the scan before or after the ends of the part originating from fixture parts are rejected. Signals occurring deeper in the part than one radius are also rejected to avoid duplication since these are picked up after 180-degree rotation of the billet.

The output of this operation is a magnetic tape record compatible with a CDC 6600 computer where further analysis is accomplished. In addition, an optional teletype print-out of all data is available.

FINAL DATA ANALYSIS

The primary test system just described acquires data which, if acquired by hand scanning, would require many man-hours and provide less precise data. System justification is also enhanced by the ease in which the data can be inserted into the CDC 6600 computer for analysis.

Analysis proceeds in two phases. First, a number of piece-part positions within the billet are examined and the coordinates of the best of these are delivered to the requestor for his selection. Second, in order to aid the requestor in his selection, histograms of the flaw distribution are drawn and orthogonal views of the flaws for 3-D simulation are drawn. This latter capability was added a few months after the initial program was instituted.

Piece-Part Location

In order to obtain piece parts, in this case nose tips (Fig. 11), with the optimum properties, a careful analysis of all possible positions within the billet must be made. The hollow nose tip is a cone approximately 10 inches long, 4 inches in diameter at the base, and 0.4 inch in wall thickness. In some cases, the cone may be truncated. The billets are usually 8 inches in diameter by 11.5 inches long.

A Fortran program has been written which, with some constraints, analyzes all possible cone positions. The major constraints are as follows:

- . No part of the cone will intersect the outer 1/8 inch deep skin of the billet.
- . No part of the cone will intersect material within 1/4 inch of the ends of the billet.
- . The axis of the cone will be parallel to the billet axis.

Since there are still an infinite number of possible positions, certain increments are established by the requestor each time a computer run is started.

Perhaps the easiest way to explain the program operation is by example (Fig. 12). The first location to be examined is with the axes of the cone and billet coincident and with the base of the cone at a specified distance, say, 1/4 inch from one end of the billet.

The computer evaluates that location and assigns a weighting factor. The cone position is translated out along a radius to give ΔR . This location is evaluated and weighted. Now the cone axis is moved along the circle with radius ΔR for a selected angular increment, $\Delta \theta$. Weighting is again performed.

When that circle is circumscribed, the radius is increased to $2\Delta R$ and the process repeated around that circle. It is desired, however, to retain an equal-volume relationship. Therefore, each time the radius is increased by ΔR , the angular increment is cut in half; e.g., at radius = ΔR , $\Delta \theta = \Delta \theta$, at radius = $2\Delta R$, $\Delta \theta = \Delta \theta/2$, and at radius = $3\Delta R$, $\Delta \theta = \Delta \theta/4$, etc.

At a certain $N\Delta R$, the sum of $N\Delta R$ and the cone radius is equal to or greater than the radius of the billet minus 1/8 inch. In other words, the cone intersects the 1/8 inch billet skin. Now, the process is halted, the base of the cone is incremented (ΔA) down the billet axis, the axis of the cone is again made coincident with the billet axis, and the process is repeated.

After several increments, $N\Delta A$, down the axis, the tip of the cone intersects the 1/4 inch skin on the other end of the billet. The process is again halted, the cone is reversed with respect to the billet, and the entire process is repeated.

By selection of the ΔR , $\Delta \theta$, and ΔA , the requestor may require the computer to evaluate any desired locations. Depending on the number of flaws, this usually varies from 200 to 2000 positions. The more flaws, the greater the number of positions necessary for optimum selection.

As the evaluation proceeds, the computer retains a number of these positions, say, 5, 10, or 15 as selected by the requestor, which have the lowest weighting factor. At the end of the run, these positions are printed out along with the four parameters describing each flaw included in the cone volume.

Cone weighting factors are selected by a repetitive weighting of each flaw with respect to the cone volume in each position (Fig. 13). In assigning the weight to each flaw, two factors are considered: (1) the amplitude of the echo from that flaw, and (2) the position of that flaw with respect to the wall thickness. That is, each flaw, which, according to its position coordinates, will be included in the wall of the cone is assigned a weighting factor, f , = 1. Weighting factors are assigned to flaws near the cone in a linearly decreasing manner. Then the weighting factor is multiplied by the echo amplitude, say, 60 percent, to assign the weight to that flaw, and summing the flaw weights results in a total weight for that cone position. That is, $f \times A = w$ and $\Sigma w = W$.

A clustering parameter and a crack potential factor are also computed. The clustering parameter counts the number of flaws within a certain radius, r , of each flaw. A similar, but different number results in a crack potential factor. Imagine all flaws connected to all other flaws by lines. Counting the lines equal to or shorter than r helps the requestor in selecting the cone location.

Heretofore, we have been concerned only with conical shapes; however, the program is adaptable to at least six configurations. These are hollow or solid cones, truncated cones, and cylinders (Fig. 14). Any size piece part may be

considered. For example, a recent application involved truncated cones approximately 3 inches long and 3 inches in diameter. Since the billets are 8 inches in diameter by 12 inches long, it was desired to cut more than one part per billet. The program was run in the normal fashion retaining the 15 best positions. Since these positions were not exclusive, the program was run again. This time, however, the billet was theoretically cut in half which yielded two right cylinders with 15 positions retained for each half. A third run was made, with the billet "cut" in thirds which yielded 45 more positions. Of the 90 positions thus generated, some of which were duplicated, it was possible to pick the desired 6 unique locations for piece parts.

While a million or more calculations are involved in a billet with 2000 to 3000 flaws and many possible nose-tip positions, the speed and memory capacity of the CDC 6600 computer reduces the operation to a matter of seconds.

In certain cases, after a cone location has been selected, it may be desired to select the best of the "scrap" material for fabrication of auxiliary parts. By searching for the "best" cylinder of small size not intersecting the cone, this is easily accomplished.

Data Presentation Displays

When the requestor receives the positions determined by the former program as having the lowest weight, he is still faced with the problem of selecting the actual position at which the cone or other part will be machined. To help him in that selection, two other computer outputs are prepared.

Data Histograms

In order to show any bias of the location of flaws within the billet and thus reveal nonuniform manufacturing techniques, histograms (Fig. 15) are prepared which display the number of flaws in a given segment of the billet. Three histograms are prepared which give the number of flaws, in percent of the total, as functions of angle from the centerline, length from the end, and radial distance from the center in equal-volume increments.

Angular distribution is calculated at some angular increment (i.e., 36 degrees), yielding ten bars on the histogram. Length down the billet is usually broken into ten parts also. Such divisions provide equal-volume increments. However, dividing the radius into equal increments does not yield equal volumes, and so, in order to do this, the radial divisions were established as follows where the volume of a cylinder is proportional to the area.

Divide the cross-sectional area of billet, A_B , by 10

$$\frac{A_B}{10} = \Delta A \approx \text{const}$$

$$\text{Area of cylinder} = \pi(R_L^2 - R_S^2) = \Delta A$$

where

R_L = radius, large

R_S = radius, small.

Then

$$R_L = \sqrt{\Delta A/\pi + R_S^2}.$$

For the first computation,

$$R_S = 0$$

$$R_{L_1} = \sqrt{\Delta A/\pi}$$

while in the second

$$R_{L_2} = \sqrt{\Delta A/\pi + R_{L_1}^2}$$

and, for the next,

$$R_{L_3} = \sqrt{\Delta A/\pi + R_{L_2}^2}.$$

Thus, ten equal-volume increments are created and plotted as histograms.

3-D Data Plots

Another aid to the requestor is 3-D data plots showing each flaw position in the billet. A true isometric or similar view could become cluttered beyond comprehension for a billet with a large number of flaws. Since spatial viewing was desirable, a means of accomplishing this was devised.

First, the billet was "cut into pieces" by the computer (Fig. 16), and then the pieces were displayed in orthogonal views by a Stromberg-Carlson 4020 plotter. Slabs were cut parallel to the billet axis and the 0 to 180 degree plane -- four slabs on the left (270 degrees) and four on the right (90 degrees) running from end to end on the billet and numbered 1, 2, 3, 4 from the center to the outside. Slices were cut perpendicular to the axis (parallel to the end surfaces). Usually, eight slices are cut.

The plotter prints, in series, the outline of the left slabs 4, 3, 2, 1, showing all flaws located in each individual slab with division lines indicating the slices, then the eight circular slices with all flaws in each slice and division lines shown for the slabs, and then the right slabs are printed. Examples of a slab and slice print-out are shown in Fig. 17A&B.

Grid lines were spaced at each 1/2 inch so that dimensions could be established as marked by small numbers on the plot. Each slice and slab was identified and the overall dimensions were given. A simple comparison of the views (Fig. 18) will locate any flaw in its spatial relationship to the billet. Clustering of flaw indications is obvious.

A code disclosing the amplitude range of each flaw echo was adopted:

<u>Symbol</u>	<u>Percent Full Scale</u>
O	0 - 20
U	20 - 40
X	40 - 60
⊗	60 - 80
*	80 +

It is possible to have echo amplitudes greater than 100 percent of full scale. Normally, if the echo from a 0.030 inch diameter flat-bottomed hole is adjusted to 40 percent of full scale, the echo from a 0.060 inch diameter equivalent flaw will be more than 2-1/2 times as high or greater than 100 percent on the CRT. While the Immerscope CRT will not show the maximum amplitude, the peak-readers will generate a voltage equivalent to the maximum.

DISCUSSION OF RESULTS

As of this point in the program, 21 billets have been evaluated by the system. Of these, four were analyzed for location of multiple small cones rather than one large cone. Some of these billets were AXF-5Q graphite made by the Poco Graphite Company. To truly judge the results, one should look at the success or failure of the end product.

Several nose tips have been cut from the billets as prescribed by this test system. Two full-scale reentry vehicles, using nose tips selected by the system, were exposed to moderate reentry conditions at the Pacific Test Range. The flights were completely successful. Other nose tips have been subjected to other types of environmental tests and these also successfully withstood the load.

Prior to the construction of the scanning system, the billets were manually scanned and the data were manually recorded. Punched cards were then prepared and fed into the computer. On one billet, the hand-scanning operation required over 40 manhours with the average about 8. Two to four hours were required for card preparation. With the new system, initial setup and calibration require about one hour, but subsequent setups require only a few minutes. Seven billets were tested in one day, dramatically illustrating the time-savings possible with this system. Even more important is the increased precision possible with the automatic process as compared to hand scanning and measurement.

Another improvement realized with this system is the capability of handling a higher sensitivity test. While ultrasonically scanning a billet, a certain "noise" level appears on the CRT which results from smaller and smaller interfaces in the material. Operator fatigue and discrimination capabilities limited the number of these which could be measured to about 1000. With the new system, this number is greatly increased and has been used to record over 11,000 discrete points (some of these were multiple indications of the same flaw). The actual number is not as important, however, as the precision in location and amplitude measurements.

The reliability of finding significant flaws manually was not measured but is undoubtedly rather low. With this system, however, the reliability is well over 99 percent (0.99).

CONCLUSIONS

The ultrasonic data system has been in operation for several months and certain conclusions may be drawn.

- . The system primary test provides a rapid and accurate method of acquiring and recording ultrasonic data.
- . The final data analysis system provides a rapid and accurate means of evaluating test data.
- . Visual aids, such as histograms and 3-D presentations, are valuable in reaching decisions concerning the product.
- . The system can be adapted to many materials and many piece parts.
- . The automation and computerization concepts can be applied to even more tests and products.

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Fig. 1. ATJ-S Graphite Billet.

Fig. 2. Block Diagram, Ultrasonic Test System.

Fig. 3. Immersion Tank and Bridge Assembly.

Fig. 4. Rotating Fixture with Billet.

Fig. 5. Search Tube, Manipulator, and Bridge.

Fig. 6. Bridge Control Panel.

Fig. 7. Ultrasonic Tester.

Fig. 8. Signal Interface System (Temporary).

Fig. 9. Block Diagram, Signal Interface System.

Fig. 10. On-Line Computer System.

Fig. 11. Graphite Nose Tips in Rough Machined Condition.

Fig. 12. Relative Cone Positions, Showing First, Second, and Third Positions.

Fig. 14. Six Normal Piece-Part Configurations, All Circular Cross Sections.

A Angle Increment Total Count 3478		B Length Increment Total Count 3478		C Equal Value Total Count 3478	
Dimension Limit	Percentage	Dimension Limit	Percentage	Dimension Limit	Percentage
30.00	11.96***	1.56	11.27***	1.24	29.56***
60.00	5.64***	2.62	14.49***	1.75	10.41***
90.00	9.55***	3.68	12.13***	2.14	10.03***
120.00	7.45***	4.74	8.40***	2.47	10.06***
150.00	6.90***	5.80	6.93***	2.76	8.40***
180.00	6.56***	6.86	5.89***	3.03	7.68***
210.00	5.18***	7.92	11.47***	3.27	5.03***
240.00	9.23***	8.98	11.07***	3.50	6.01***
270.00	7.39***	10.04	9.55***	3.71	8.83***
300.00	7.62***	11.10	8.80***	3.91	4.00***
330.00	8.65***				
360.00	12.94***				

Fig. 15. Computer-Plotted Histograms: (A) Angle Increment, (B) Length Increment, and (C) Equal Volume.

Fig. 16. Slice and Slab Generation System.

Fig. 17A. Typical Slice Computer Plot.

Fig. 17B. Typical Slab Computer Plot.

Fig. 18. 3-D Flow Correction.

APPENDIX A

Preparation of Billets and Calibration

Procedure for the Immerscope - H-21 Computer System

The ATJ-S billets are first mechanically measured and the dimensions noted. The billets have serial numbers scribed on one flat end and this becomes the reference or base end of the billets. An arbitrary point is chosen on the reference end of the billet and is marked as 0 degrees. The angular position of the billets increases in a clockwise direction looking at the reference end.

A polyvinyl chloride coating is next sprayed on the billets, thus providing a strippable layer to protect the billets from water. In addition, the PVC coating provides a suitably smooth surface for ultrasonic inspection.

The H-21 computer provides four pieces of print-out information about each defect within the billet. These are:

1. Flaw amplitude in percent of full scale.
2. Depth of flaw in inches.
3. Angle of part in degrees.
4. Position of flaw from left or reference end in inches.

The calibration procedure consists of giving the H-21 the necessary data, in the form of DC voltages, for calculating the four pieces of information.

The DC voltages for calibrating the first two items above (flaw amplitude and depth) are static simulations of what, in the actual test, are dynamic occurrences. The other two items (angle of part and flaw position) are interrogations of DC voltages on potentiometers that are proportional to angle and horizontal position.

The flaw amplitude calibration voltage is obtained by putting a flaw signal on the Immerscope CRT that is 100 percent full scale. The amplitude channel of the interface is now applied to an auxiliary oscilloscope and a DC voltage, generated by a variable DC power supply, is inserted into the H-21 that is equal to the dynamic DC signal generated by the sample and hold circuitry. The H-21 now has a reference for flaws of 100 percent amplitude and during billet inspection will compare all flaw levels to this reference and print out data proportional to this reference.

The depth of flaw is obtained in much the same manner. The H-21 is told the radius (in inches) of the specimen and is then given a DC voltage that simulates that radius. The H-21 will now compare all flaw signals received from the interface to the known reference for the radius of the specimen and place them in the proper position, depth-wise, in the billet.

The angle of the part is obtained by interrogating a rotation potentiometer connected to the end of the billet. The calibration consists of inserting into the computer the voltage equal to 360 degrees. The H-21 program, as written, is limited to calculating the angle from 0 to 360 degrees; therefore, 0 degrees on the billet must be aligned to correspond to 0 volts on the potentiometer.

The position of the flaw from the left, or reference end, is obtained by interrogating the potentiometer located on the immersion-tank scanning fixture. The length (in inches) of the specimen is inserted into the computer. The difference between two DC voltage levels, set by manually positioning the ultrasonic transducer over both ends of the specimen, is then proportional to the length of the billet.

Initiating the H-21 computer data entry program provides a print-out on the teletype as illustrated on Fig. A1.

Step-by-step procedure for calibrating the system under computer control is as follows:

1. Type in length of specimen.
2. Type in radius of specimen.
3. Bring into the computer, through an A/D converter, the following:
 - a. Far (right) limit (EOPR on Fig. A1) by positioning the ultrasonic transducer over far (right) end of specimen, using Immerscope CRT as a reference.
 - b. Left (near) limit (EOPL on Fig. A1) by positioning the ultrasonic transducer over the left (near) end of the specimen, again using the Immerscope CRT as a reference.
 - c. Radius of specimen (MAXD on Fig. A1) using a dual-channel oscilloscope with the video from the Immerscope on channel one input and the sweep interval from the Immerscope on channel two input. With the Immerscope sweep controls, set front and back ultrasonic echoes of the billet on the oscilloscope. Measure the transit time of the ultrasonic signal through the billet. Now, the Immerscope sweep controls are set so that sweep interval on the oscilloscope is $1/2$ the transit time through the billet. The inputs to the oscilloscope are then disconnected and replaced by D1 (first distance channel) of the interface and an external DC power supply. The DC power supply is then set to match the level of D1. Note: Caution must be taken so that no flaws are present on the Immerscope CRT. With the power supply thus set, disconnect from oscilloscope and insert this level into the computer where it is sampled and stored.
 - d. Reading for 360 degrees (ORA 360 degrees on Fig. A1) by rotating the billet until maximum voltage appears on the rotation potentiometer. This voltage is then inserted into the computer where it too is sampled and stored. During testing, the voltage should increase as degrees on the reference end of the billet increase. After the above is done, the potentiometer linkage is loosened and the billet is aligned with the 0-degree reference at 0 volts on the potentiometer.
 - e. Reading for maximum amplitude expected (ORFSAM on Fig. A1). This is done by using a dual-channel oscilloscope with A1 from the interface connected to channel one and a DC power supply connected to channel two. First, ultrasonically locate a known flaw in an ultrasonic reference block and set the Immerscope gain so that the flaw signal is 100 percent of full scale on the CRT, while making certain no other flaw is present between the front surface echo and the

known flaw. Then, set the DC power supply voltage to equal the A1 interface amplitude signal. After the power supply voltage is set, connect it to the H-21 computer where it is sampled and stored.

The above procedure has now given the H-21 the necessary information to allow it to assign to each flaw received a depth within the billet, a distance down the billet from the reference end, a rotational degree location around the billet, and an amplitude which is a relative measure of its size.

The final calibration step is to set the overall sensitivity of the entire system. As mentioned in the introduction of this report, the ATJ-S billets contain many discontinuities. By using the H-21 computer for on-line, real-time analysis and data storage, inspection time is no longer the limiting factor. The amount of data available is now the only test parameter considered. By increasing the sensitivity of the system, enough data can be obtained that the time needed to process the data on the large computer (CDC 6600) becomes prohibitive.

At first glance, the most obvious place to change the sensitivity is the Immerscope gain. This gain cannot be changed now, however, because, as shown in the calibration procedure, this gain is set to known flaw levels and the H-21 assigns flaw levels accordingly. Provision for adjusting sensitivity is provided on the interface. This sensitivity determines the level (percent of full scale on Immerscope CRT) at which the interface will start to accept flaw data. While watching the Immerscope video signal on one channel of an oscilloscope and the flaw interrupt signal (TP3 on card 10 of interface) on the other channel, the sensitivity control on the interface is varied to determine the level at which the flaw interrupt signal will start.

The ATJ-S billets, at the Immerscope gain settings mentioned, have an overall baseline signal, or noise, of 25 to 30 percent of full scale. Therefore, the interface sensitivity control is set so that Immerscope flaw signals between 30 and 40 percent initiate a flaw interrupt to the computer. The above sensitivity level determines the number of data points processed by the computer and stored on magnetic tape. On one comparatively "noisy" billet, 11,493 flaws were recorded when the flaw interrupt sensitivity was set at 30 percent. By reducing the sensitivity so that 40 percent flaws started the interrupt signal, the number of flaws in the same billet was reduced to 3,156. This vividly illustrates that most of the flaw information in ATJ-S billets is near the noise or background level.

The Immerscope gain and sensitivity time control (STC) are set so that a 0.031-inch diameter, flat-bottom hole located 1.0 inch below the surface of ATJ-S material is 80 percent of full scale and the same size flaw at 3.0 inches below the surface is 40 percent of full scale. Knowing these levels for known flaws places some meaning on amplitude readings for flaws from billets which cannot be destructively analyzed or test-drilled with known size holes. The differing responses to the above known flaws vividly illustrate the high attenuation of the ATJ-S material.

ENTER THE FOLLOWING CONSTANTS ACCURATE TO NO MORE THAN + OR - .01

THE SPECIMEN LENGTH IN INCHES = 12.50	14.37	IN OCTAL
THE SPECIMEN RADIUS IN INCHES = 2.55	02.43	IN OCTAL

YOU WILL NOW GET A HALT ON THE COMPUTER. POSITION THE ULTRASONIC ARM OVER THE FAR (RIGHT) END OF THE SPECIMEN AND THEN GO TO "STEP AND RUN" ON THE COMPUTER.
EOPR = 2174

REPEAT THE ABOVE INSTRUCTIONS FOR THE LEFT (NEAR) SIDE OF THE SPECIMEN
EOPR = 1462

STEP-RUN TO BRING IN THE OCTAL READING OF THE RADIUS OF THE SPECIMEN.
MAXD = 1210

STEP AND RUN TO BRING IN THE OCTAL READING OF 360 ON THE ANGLE POT.
ORA360 = 1765

STEP AND RUN TO BRING IN THE OCTAL READING OF MAX AMPLITUDE EXPECTED.
ORFSAM = 1516

Fig. A1. H-21 Computer Data Entry Sheet.

APPENDIX B

Real-Time Data Presentation

An example of the real-time print-out as obtained from the teletype is illustrated on Fig. B1. The print-out is controlled by the H-21 computer. The print-out can be initiated or halted any time during the ultrasonic test by operation of a switch on the H-21. The print-out contains all the processed data in engineering units and in the actual readings which are recorded on the magnetic tape.

The first column, Data Block Length, indicates the number of data words processed and recorded on one axial sweep of the ultrasonic transducer over the billet. This number is recorded on the magnetic tape in octal notation along with the octal data words. Since each flaw requires four data words to describe its location and amplitude, dividing the data block length number by four will give the number of flaws for one scan.

The second column, Flaw Level Number, indicates the number of flaws at one instant under the transducer looking through the radius of the billet. It is possible for several flaws to be lined up one on top of the other at one point in the billet. When this occurs, it is highly desirable to be able to record each of the flaws. The electronic interface used in this system is capable of processing information from five flaws at different depths at one instant. The operation of the interface is covered in detail in Appendix C. The print-out of flaw level is as illustrated. When only one flaw is received, only flaw level one is used. When more than one flaw in depth is received, successive flaw levels are used and are indicated by the print-out of two, three, etc., for as many flaws as received up to a maximum of five. The print-out must be in successive ascending numbers, if not it is an indication of a malfunction in the system.

The remaining four columns are the amplitude, depth, angle, and position of the flaws as illustrated. Observation of the four parameters of the flaws is a very useful diagnostic tool. Any unusual deviation in any of the columns is cause for stopping the test. At a glance, it can be seen whether or not the billet is incrementing properly. Depth of flaw and position of flaw are good indicators of proper calibration. Unusually high or low amplitude readings would be scrutinized closely.

At the end of the inspection of the billet, the H-21 computer will type out the message as illustrated on Fig. B1. This is a summary of the complete test and is especially useful for a quick qualitative comparison of one billet to another. Here again, dividing the number of data words by four will give the total number of flaws processed and recorded during the ultrasonic examination of this particular billet. The total number of sweeps can be varied, as desired, and is listed to show how many sweeps were used.

1-----ENGINEERING UNITS-----1 1----OCTAL READINGS---1

DATA BLOCK LENGTH	FLAW LEVEL NO.	FLAW AMPLITUDE IN % F/S.	DEPTH IN INCHES	ANGLE OF PART	POSITION OF FLAW FROM LT.	AMP.	DEP.	ANG.	POS.
12	1	69.24	1.44	0.00	5.62	1072	0702	0000	1256
	1	68.63	2.62	0.00	4.26	1065	1206	0000	1157
	1	57.94	0.70	0.00	0.36	0735	0335	0000	0650
8	1	73.13	0.00	1.76	1.74	1123	0000	0005	0731
	1	52.46	2.24	1.76	12.26	0660	1274	0005	1747
	1	64.74	1.96	20.00	0.13	1025	1152	0070	0654
	1	104.12	2.25	20.33	1.87	1531	1300	0071	0776
	1	60.02	1.20	20.33	4.44	0756	0570	0071	1167
	1	76.95	2.52	20.00	6.37	1151	1421	0070	1322
	1	54.53	0.53	20.33	8.43	0701	0255	0071	1462
12	1	53.81	2.50	180.00	2.57	0673	1200	0770	1037
	2	53.81	2.24	180.00	2.57	0673	1165	0770	1037
	1	74.83	2.52	180.00	6.46	1150	1417	0770	1326
12	1	85.76	0.63	360.00	6.55	1302	0311	1762	1332
	1	70.70	1.44	360.00	5.57	1106	0706	1762	1254
	1	50.41	1.31	360.00	4.24	0637	0630	1762	1155

TEST OVER. HALTING OPERATION.
TOTAL NUMBER OF SWEEPS FOR THIS BILLET IS 360.
TOTAL NUMBER OF DATA WORDS IN ENTIRE TEST 4180.

Fig. B1. H-21 Computer Flaw Record.

After a few billets were inspected using the Immerscope-interface-H-21 computer system and it became known that the system was operating properly, it was observed that a 100 percent real-time print-out by the teletype of the flaw data from one billet would require too much time. It was not uncommon to require two to three hours to completely inspect one billet and print out the data. In comparison, 15 to 30 minutes were required if the print-out was deleted.

APPENDIX C

Operation of the Interface

This appendix describes the operation of the computer interface used in the Sandia-built ultrasonic test system. Operating as a link between the Immerscope 424D and H-21 computer, this device must convert fast-pulse, ultrasonic flaw signals into a form compatible with the H-21. It is insufficient to know only that a flaw exists; its amplitude and depth must also be known. Since it is possible to receive more than one echo associated with any one excitation pulse, it also becomes necessary to know how many echoes there are. These, then are the requirements of the interface:

1. Determine how many, if any, flaw echoes have been received.
2. Measure their individual amplitudes.
3. Measure their individual depths as a function of time.
4. Inform the computer that the echoes have been received and make all of the above information available in voltage form at the right time.
5. Perform all of the above for each repetition of the excitation pulse (200 times every second).

The location of the transducer over the test part is also necessary information; however, angular and distance coordinates are continuously available from position transducers which are automatically interrogated any time there is flaw information. Since this is a program operation, it is not considered part of the interface.

The interface has the capacity to measure five flaws in any one frame, where a frame is that period of time associated with any one excitation pulse. A detailed description of this interface follows.

Immerscope Adjustment

Refer to Fig. C1. Trace A. This is the overall timing for the ultrasonic system. The first echo seen after the excitation pulse is the front surface. Any flaws in the material would come next, then the back surface echo. This entire series repeats every 5 milliseconds, or 200 times per second. Because the transducer is in motion, each frame is "viewing" a slightly different location. The delay and sweep controls on the Immerscope are adjusted so as to display, on the CRT, only that portion of time that is of interest. This portion of time extends from just before the front surface to just beyond half way through the part and is depicted in Fig. C1, Trace B, as the sweep interval. The other half of the cylinder is tested as the part is rotated. This time interval is very important because the echoes from any flaws in the material

must occur during this time. By agreement, the first echo on the CRT shall be the front surface; that is, the first signal in time during the sweep interval shall be the front surface. This is necessary to satisfy all the logical requirements that follow.

Peak-Readers

The peak-readers used in this system are custom-made and must respond to very fast signals (less than 100 nanoseconds rise-time). These readers respond only to positive signals and will retain the most positive input seen since the last time they were reset to zero. Their inputs may be gated and their outputs inhibited at will. They may also be reset at any time. Ten of these peak-readers are used in the interface: five read echo pulse amplitude and five read echo time or depth. The function of the logic modules is to steer the proper input and gating signals to the ten peak-readers. This may now be considered in some detail.

The Depth Ramp

Figure C2, Trace A, shows a typical CRT display with a front surface echo followed by four flaws. The sensitivity threshold for the interface is determined by a Schmitt trigger and is represented by the dotted line shown in Trace A. As can be seen, the fourth echo is of insufficient amplitude to be considered. Trace B is a pulse representative of the sweep interval. Trace C is the Schmitt trigger output.

If the velocity of the material being tested is assumed to be constant, then the distance a flaw is below the front surface is directly proportional to time. To measure this time, a linear ramp is started with the receipt of the front surface echo and terminated at the end of the sweep interval. This ramp generation time is shown in Trace D and is derived from logical operations using B (sweep time) and C (Schmitt trigger output). The ramp itself is shown in Trace E. This ramp is simultaneously applied to all five depth peak-readers. When the first flaw echo appears, the input to the channel one depth peak-reader is inhibited; that is, the ramp at its input is cut off. As each successive flaw appears, their respective inputs are inhibited. This action is shown in Fig. C3. The peak value attained at each input will be held by the peak-readers until they are reset.

The acceptance time for the ramp at the various inputs is shown in Fig. C3 and at the bottom of Fig. C2. Figure C4 shows how these ramp acceptance times are derived. The Schmitt trigger pulses are fed to a BCD counter and the counter output is decoded as shown for the first three channels. These decoded intervals are gated with the original Schmitt trigger pulses to create the ramp inhibit pulses. These inhibit pulses reset flip-flops which were set by the front surface signal. The output from these flip-flops gates the ramp off and on at the input of the peak-readers. Notice again that the example shows no fourth and fifth inhibit pulses.

These inhibit pulses, operating through one-shots (monostable multivibrators), create the interrupt pulses that tell the computer which flaws have been received and, consequently, which channels to interrogate. Although channels four and five receive completely uninhibited ramps, their outputs will not be interrogated because there are no flaws to generate interrupts.

Amplitude Peak-Readers

The five amplitude peak-readers operate in much the same way. The decoded counter output used to determine when to inhibit the ramp inputs to the depth peak-readers is also used to gate the inputs for the amplitude peak-readers. This time, however, no further logical operations are necessary and the decoded intervals are used in their existing state.

The video signal is simultaneously applied to all five amplitude peak-readers. The counter outputs hold the various gates open only long enough to pass the appropriate pulse to the appropriate peak-reader. The effects of this operation are shown in Fig. C5. The amplitudes of the echoes will be held by their respective peak-readers until they are reset.

Output

A pictorial representation of the operation of all the peak-readers is shown in Fig. C6. The actual final output of the peak-readers is inhibited (held at zero volts) until the end of the sweep interval. At that time, the outputs are allowed to rise and remain so for approximately 0.7 millisecond, and then all ten of the peak-readers are reset simultaneously by a 0.16-millisecond pulse. The timing for these events is shown in Fig. C1.

During the 0.8-millisecond interval, the computer interrogates the interface for information. When a channel one flaw interrupt is received, its amplitude is immediately interrogated, followed by its depth, then its coordinates. This information is processed, then stored in the computer's memory. The computer then looks to see if a channel two interrupt has been received. If it has, all channel two information is interrogated in a similar manner. If necessary, this process continues for all five channels. The computer then resets its interrupt lines and waits again for a channel one interrupt.

After the transducer scans off the part, an end-of-part interrupt is generated which instructs the computer to process and output all the information stored for the entire scan. During this output time, the computer inhibits the scanning mechanism as long as necessary to place all the information on tape. At the present time, this interrupt is generated by an adjustable mechanical switch assembly mounted on the scanning fixture. A later modification will derive the interrupt from the front surface echo; that is, as the transducer leaves the part, the front surface echo disappears.

Fig. C1. Ultrasonic System Overall Timing Diagram.

Fig. C2. Timing Diagram, Depth Peak-Reader.

Fig. C3. Ramp Inhibit Timing Diagram.

Fig. C4. Ramp Acceptance Timing Diagram.

Fig. C5. Gate Timing for Amplitude Peak-Readers.

*THE FINAL OUTPUT OF THE PEAK-READERS IS INHIBITED UNTIL THE END OF THE SWEEP INTERVAL (CRT DISPLAY TIME).

Fig. C6. Peak-Reader Outputs.

CONSTITUTIVE EQUATIONS

Chairman: *C. E. Pugh*, Oak Ridge National Laboratory

MATHEMATICAL REPRESENTATION OF MECHANICAL BEHAVIOR
OF NONLINEAR INELASTIC MATERIALS

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ABSTRACT

At elevated temperatures and stresses, the present behavior of a material would depend upon past temperatures and stresses to which the material has been subjected. The mathematical representation of this dependence upon past is the subject of this lecture.

Of the two basic modes of representation, the one which is based on state variables and differential equations is often more convenient and physically more meaningful.

It will be shown that by studying the results of appropriately planned phenomenological experiments, one could obtain information on the nature of the state space which constitutes the foundation of this mode of representation.

1. INTRODUCTION

Rational analysis of structures subjected to elevated temperatures and stresses demands that mechanical behavior of materials which constitute the structure should be well understood and should be represented with desirable accuracy by mathematical means. Under elevated temperatures and stresses most materials of interest exhibit nonlinear, time-dependent behavior. The description of such behavior is a many-faceted problem. Although the physical basis of deformation processes in metals is rather well understood, there is yet no satisfactory way of incorporating this knowledge into the stress-strain relations and thermodynamics. From this point of view phenomenological tests constitute a more readily accessible source of knowledge. The present paper is an attempt to review the methods

available for transferring experimental knowledge into constitutive relations.

In order to keep the notation and mathematics at a simple level, we confine ourselves to a one-dimensional situation and consider isothermal deformations of a bar subjected to uniaxial time-dependent stress by means of a conventional testing machine. For a generalization of these ideas to the three-dimensional case, the reader is referred to references 1 and 2.

The paper starts with a consideration of a set of experiments performed with aluminum at a moderate temperature. The nature of nonlinearities that this material exhibits is discussed.

It is next indicated that the integral representation of mechanical behavior is not suitable for most metals. Attention is then focused on the mode of description based on state variables. It is shown that phenomenological tests can be used to obtain information on the number of state variables and on the equations which govern these variables.

2. CREEP OF A TYPICAL METAL UNDER TIME-DEPENDENT STRESS

We comment here briefly on the results of creep experiments by Wang and Onat³ conducted with 1100 aluminum at 300°F. We do so in the hope of showing that creep is a highly non-linear phenomenon and that non-linearity of creep lets itself be felt in many ways. It will be apparent that most creep laws used in the engineering literature (for a thorough survey of creep laws, see Rabotnov⁴) cannot describe accurately types of non-linear phenomena observed in the above-mentioned tests.

Figure 1 describes rather schematically the results of several tests which involve the application of the uniaxial stress $\sigma(\tau)$, where τ denotes time, and the observation of the resulting strain $\epsilon(\tau)$ in several initially identical test specimens.

Tests 1 and 2 are ordinary creep tests. These show that in 1100 aluminum creep does not occur below a certain stress level; at higher stress levels creep occurs, the rate of the steady creep being a highly non-linear function of the stress intensity.

Tests 3 and 4 constitute two different continuations of 2. Test 3 shows that under incremental loading an appreciable transient creep

develops. On the other hand we see from test 4 that incremental unloading is accompanied by a sudden elastic response followed by slowly increasing creep deformation. There is clearly asymmetry in behavior with respect to incremental loading and unloading, which is reminiscent of and related to the plasticity of metals.

Test 5, which may be called an interrupted creep test, exhibits features which could be found only in non-linear materials. These are: (a) there is a lack of strain recovery (except the sudden elastic one) upon complete unloading, in spite of the presence of transient creep in a creep test; (b) the transient creep observed upon reloading is weaker than the transient creep obtained at the beginning of a creep test.

The present paper is devoted to a discussion of mathematical representation of stress-strain relations for materials such as 1100 aluminum.

3. MATHEMATICAL REPRESENTATION OF CREEP BEHAVIOR

Each test of the type discussed in the previous section provides one with a pair of functions $\epsilon(\tau)$ and $\sigma(\tau)$. If the test machine is such that the strain $\epsilon(\tau)$ is prescribed by the experimenter and $\sigma(\tau)$ is obtained as the test result, then one may say that the material responded to the input $\epsilon(\tau)$ and produced the output $\sigma(\tau)$. In the terminology of system theory the test specimen or the material acts as a black box and assigns outputs to given inputs. The task of the engineer is to understand this input-output relationship and then to represent it mathematically.

For inelastic materials, such as creeping metals, this relationship is a complicated one. The present stress is no longer a function of the present strain, as it would be in an elastic bar, but it depends on happenings in the past, namely, on previous deformations of the bar. Thus for a given inelastic material we have, in the present uniaxial case,

$$\sigma(t) = F[\epsilon(\tau)]_{\tau=0}^{\tau=t} \quad (1)$$

where $\sigma(t)$ denotes the stress at time t . The right-hand side of this equation expresses the fact that $\sigma(t)$ depends on the history of strain on the time interval* $0 \leq \tau \leq t$. The mechanical behavior of a bar of given material is then defined by means of a particular functional F .

It is well known⁵ that when the material is linear and non-aging,⁶ then F has the following form:

$$F[\epsilon(\tau)] = \int_0^t E(t-\tau) \dot{\epsilon}(\tau) d\tau \quad (2)$$

where $E(t)$ is the relaxation modulus of the material which, in principle, can be determined by means of phenomenological experiments.⁷ If the material is non-linear, then the representation of F becomes more complicated. Nevertheless, as known since the beginning of the present century, under certain smoothness assumptions the non-linear functional F can be represented, to any degree of accuracy, by a sum of multiple integrals:

$$F \approx \int_0^t E_1(t-\tau_1) \dot{\epsilon}(\tau_1) d\tau_1 + \int_0^t \int_0^t E_2(t-\tau_1, t-\tau_2) \dot{\epsilon}(\tau_1) \dot{\epsilon}(\tau_2) d\tau_1 d\tau_2 + \dots \quad (3)$$

where for the sake of simplicity only the first two terms are shown. If Eq. (3) is to be used for the representation of mechanical behavior, then a sequence of increasingly complicated kernels $E_1(t_1)$, $E_2(t_1, t_2)$, etc. must be specified for a given non-linear inelastic material.

Attempts have been made by Ward and Onat⁸ and Findley and Onaran⁹ to determine experimentally the number of terms to be retained in Eq. (3) and the kernels of these terms. As was discussed in reference 6, for a class of solids which may be denoted as weakly non-linear, the above mode description is quite convenient.

*We assume that at time $\tau=0$ the bars are in the stressless, virgin state and that this state is identical for bars of a given material.

However for a broader class of materials, which include plastic or nearly plastic solids such as creeping metals, a prohibitively large number of terms in Eq. (3) would be needed for any reasonable accuracy in representation.⁶ Wang and Onat have shown in a paper³ dealing with a representation of the test results discussed in Section 1, that a modified integral representation which is based on the partitioning of the total strain into elastic, plastic, and time-dependent components is not quite successful.

Another drawback of the representation in Eq. (3) is that the kernels are not related in a transparent and direct way to the physics of the deformation. This is especially true for the class of materials mentioned above. Representation of F by means of state variables does not suffer from these drawbacks.

4. REPRESENTATION BY MEANS OF STATE VARIABLES

We adopt a microscopic point of view to introduce the state variable representation. We observe that the stress at a given time would depend on what is happening within the specimen at that time. More precisely the present stress would depend on the positions and possibly velocities of the "particles" which make up the solid.* On the other hand, since the stress is a quantity which represents an average, it would not depend on all the details of the present arrangement of "particles," but rather on a number of average quantities which define the salient and relevant features of this arrangement. For a polycrystalline bar these quantities may be the average interatomic distance in the direction of load application, the number of dislocations, other average quantities related to dislocation distribution, etc.

Let us now assume that we need n quantities, $q_1, \dots, q_n$, to specify those features of particle arrangement which are of relevance to mechanical behavior. If for two different bars we find that these features are measured by the same set of quantities $q_1, \dots, q_n$, then these specimens would

*Since the present arrangement of particles is brought about by previous deformations, it is also correct to assert that Eq. (1) holds.

carry the same stress and will behave identically, from a mechanical point of view, under identical future deformations. Let $\vec{S}$ denote a vector with coordinates q_i ($i=1, \dots, n$) in an n -dimensional Euclidean space E . We call the q_i 's the state variables and the space E the state space.

Since the stress is a function of the state of the bar, we have

$$\sigma = f(\vec{S}) \quad (4)$$

Our next task is to relate the state $\vec{S}$ to the deformation applied to the bar. For this purpose consider a bar which is in the state $\vec{S}$ at time t . This bar is subjected to further deformation

$$\begin{array}{l} \tau=t+\Delta t \\ \epsilon(\tau) \\ \tau=t \end{array}$$

on the time interval $[t, t+\Delta t]$. What would the state of the bar at time $t+\Delta t$ depend upon? In view of above remarks concerning the notion of state, the answer would be that for non-aging materials it would depend upon the state of the bar at time t and on the applied strains on $[t, t+\Delta t]$:

$$\vec{S}(t+\Delta t) = \vec{G}\left(\vec{S}(t); \begin{array}{l} \tau=t+\Delta t \\ \epsilon(\tau) \\ \tau=t \end{array}\right) \quad (5)$$

If one now takes $\Delta t \rightarrow 0$ and introduces $\dot{S}(t)$, the time derivative of $S(t)$, then one can show, under certain assumptions of smoothness, that Eq. (5) reduces to

$$\dot{\vec{S}} = \vec{\phi}(\vec{S}, \epsilon') \quad (6)$$

It can be shown that under further hypothesis of smoothness, Eq. (6) becomes

$$\dot{\vec{S}} = \vec{g}(\vec{S}) + \vec{h}(\vec{S}) \epsilon' \quad (6')$$

The above equation [(6) or (6')] may be called the growth law for the state variables. Equations (4) and (6) constitute a description of mechanical behavior. With appropriate initial conditions, and for a given $\epsilon(\tau)$, equation (6) or (6') can be integrated to find the state of the element at time t . Equation (4) then enables one to determine $\sigma(t)$.

We note that the formal structure of Eqs. (4) and (6) is very much in the tradition of classical physics. For a specific example of Eqs. (4) and (6) the reader is referred to Section 6.

The problem which faces the engineer is to discover, for a given material, the number and nature of state variables q_i and to specify the functions f and $\vec{g}$ or f , $\vec{g}$, and $\vec{h}$. To accomplish this task the engineer will make use of available experimental evidence. Some of this evidence may come from materials science and may be of microscopic nature. It may be hoped that in the description of plastic behavior of metals, the recently acquired enormous knowledge on dislocations could be used* to add substance to formal Eqs. (4) and (6).

5. PHENOMENOLOGICAL TESTS VERSUS STATE VARIABLES

Another readily available source of information is phenomenological tests. In the present case each test would involve the application of a given strain history $\epsilon(\tau)$ on some test interval $[0, T]$ and the observation of the resulting stresses $\sigma(\tau)$.

We now raise the following question: Can one, by studying the results of phenomenological tests (each result being in the form of a pair of functions $\epsilon(\tau)$ and $\sigma(\tau)$ on $[0, T]$), gain information to be used in setting up Eqs. (4) and (6) for a given material?

In order to answer this question, let us establish the connection between the notions of state and dependence on history.** For this purpose consider two distinct strain histories (1 and 2 in Fig. 2) applied on $[0, t]$

*For a recent effort in this direction, see reference 10.

**The study of this question was begun in reference 6.

to two separate but initially identical test specimens. We obtain, say, the stress responses shown in Fig. 2.

Let us assume that the two histories $\epsilon_1(\tau)$ and $\epsilon_2(\tau)$ give rise to the same stress at time t . Under this assumption we might begin to wonder whether the two specimens might be in the same state at time t . In order to investigate the situation further, we would like to apply identical future deformations to the two specimens. By identical future deformations we mean identical future strains with respect to the configurations at time t . If the strains are small this would mean that the future strains measured with respect to the configurations at time $\tau=t$ must differ from each other by the constant $\epsilon_1(t) - \epsilon_2(t)$. Figure 3 shows such an extension of $\epsilon_1(\tau)$ and $\epsilon_2(\tau)$ for $\tau > t$ (solid lines).

Suppose now that we observe the same stresses in the two test bars on $[t, T]$. Suppose further that this remains true for any extensions of $\epsilon_1(\tau)$, $\epsilon_2(\tau)$ into the future satisfying the requirement of $\epsilon_1(\tau) - \epsilon_2(\tau) = \epsilon_1(t) - \epsilon_2(t)$, $\tau > t$ (dashed lines in Fig. 3). If the above state of affairs exists then we say that the strain histories $\epsilon_1(\tau)$ and $\epsilon_2(\tau)$ on $[0, t]$ give rise to the same state at time t .

We may next inquire as to how many distinct states can be produced at time t by the application of all strain histories of interest on $[0, t]$. It may happen that the number of distinct states produced is much less than the number of distinct histories on $[0, t]$. In fact if one makes the assumption that the distinct states fill an n -dimensional space* at time t and that the material is non-aging then one obtains Eq. (4) directly from the above considerations; Eq. (6) then follows as before. We have thus indicated that the study of input-output pairs $(\epsilon(\tau), \sigma(\tau))$ is in some sense equivalent to the microscopic studies of the specimens. If such is the case, then we must be able to obtain information concerning Eqs. (4) and (6) from the phenomenological tests. Let us be more specific and inquire whether the number of state variables could be determined from the measurement of the test pairs $(\epsilon(\tau), \sigma(\tau))$.

*For more precise exposition of these ideas the reader is referred to references 1 and 2, where the general case of arbitrary deformations has been considered and questions of thermodynamics have been discussed.

For this purpose let us conduct tests where the applied strains on $[0, t]$ are piecewise linear (Fig. 4) and therefore defined by m numbers $\epsilon_1, \epsilon_2, \dots, \epsilon_m$, these numbers being the values of $\epsilon(\tau)$ at $\frac{t}{m}, 2\frac{t}{m}, \dots, t$ respectively (m and t are fixed; $m = 3$ in Fig. 4).

Now suppose that for tests of this type, each test gives rise to a distinct state. This fact, if true, can be established in principle by experiments by continuing each test beyond $\tau=t$ in the manner described in Fig. 3. Now I assert without proof that if the above situation exists then the number of state variables n is equal or greater than m , the number of linear segments in tests of the type shown in Fig. 4.

Thus we have indicated that a lower bound on the number of state variables can be found by means of phenomenological experiments.

We must note here that the above ideal procedure would require an infinite number of tests. A finite number of tests with the usual presence of scatter of data could only provide a hint that each test gives rise to a distinct state at time t .

6. MOTIONS IN STATE SPACE

In order to analyze further the results of phenomenological experiments with a view of obtaining information on the functions f and $\vec{g}$ and $\vec{h}$ which appear in the state variable representation, Eqs. (4) and (6'), it is helpful to review briefly the behavior of some simple linear viscoelastic solids in the setting of a state space. For simplicity we shall first be concerned with materials which possess only two state variables.

We consider the arrangement of springs and dashpots shown in Fig. 5 as a model of a viscoelastic material. We let c_1 and c_2 denote spring constants and μ_2 and μ_3 the viscosities of the dashpots. The force applied at point A will represent the stress, and the displacement of A will play the role of strain.

We have, then, as a result of simple calculations,

$$\sigma = c_1 q_1 \quad (7)$$

and

$$\begin{aligned} \dot{q}_1 &= -\frac{1}{\mu_2} (c_1 q_1 - c_2 q_2) - q_1 \frac{c_1}{\mu_3} + \epsilon \\ \dot{q}_2 &= \frac{1}{\mu_2} (c_1 q_1 - c_2 q_2) \end{aligned} \quad (8)$$

where q_1 and q_2 are dimensionless elongations of the springs.

In the above equations q_1 and q_2 should be interpreted as state variables, so that Eq. (7) now plays the role of Eq. (4), and Eq. (8) corresponds to the growth law, Eq. (6'), for the material under consideration.

Note that the growth law, Eq. (8), has the vectorial form

$$\left. \begin{aligned} \vec{q} \cdot &= \vec{g}(\vec{q}) + \vec{h} \epsilon \\ \text{where} \quad \vec{h} &= (1, 0) \\ \text{and} \quad g_i &= A_{ij} q_j \quad i, j = 1, 2 \end{aligned} \right\} \quad (9)$$

with

$$A_{ij} = \begin{bmatrix} -\left(\frac{c_1}{\mu_2} + \frac{c_1}{\mu_3}\right) & \frac{c_2}{\mu_2} \\ \frac{c_1}{\mu_2} & -\frac{c_2}{\mu_2} \end{bmatrix}$$

We adjoin to the growth laws, equation (8) or (9), the initial conditions

$$q_1 = q_2 = 0 \quad \text{at } \tau=0 \quad (10)$$

For a given strain history $\epsilon(\tau)$, Eq. (8) is integrated with the help of Eq. (10) to determine the state vector $\vec{q}(\tau)$ with components $q_1(\tau)$ and $q_2(\tau)$. As the strain $\epsilon(\tau)$ develops over the course of time, the end point of the vector $\vec{q}(\tau)$ describes a trajectory in state space. The first coordinate of the state point (q_1, q_2) then determines the stress via Eq. (7).

It will be desirable, for purposes of later discussions, to convert the information contained in the growth law, Eq. (8), into a geometrical form. This could be achieved by observing that $\vec{g}(\vec{q})$ and $\vec{h}$ which appear in Eq. (9) determine two vector fields in state space. These vector fields create two families of curves in state space by means of their envelopes. The family of curves created by the field $\vec{g}(\vec{q})$ will be called relaxation lines. If ϵ is kept constant during a given phase of a test, then the state point moves along a relaxation line during this phase; the motion of the point is defined by Eq. (9) with $\epsilon' = 0$;

$$\vec{q}' = \vec{g}(\vec{q})$$

or, equivalently

$$q_i' = A_{ij} q_j' \quad (11)$$

As Fig. 6 shows, the relaxation lines (of the first quadrant of the state space) converge to the origin. One relaxation line is straight. It corresponds to one of the eigen directions of the relaxation matrix A_{ij} . When the constants c_1 , c_2 , μ_2 , and μ_3 are positive and finite, A_{ij} has two real negative eigenvalues. This remark indicates that A_{ij} can be diagonalized by a suitable transformation of coordinates.

Since $\vec{h}$ is the constant vector (1,0) [cf.(9)], the family of curves created by $\vec{h}$ are simply lines parallel to the q_1 axis. These lines may be called the lines of fast deformation. As can be easily seen from Eq. (8), when ϵ' is (infinitely) large the state point moves along a line of fast deformation.

We may now discuss the images of some simple tests in state space. A relaxation test involves a suddenly applied strain which is maintained subsequently. This type of test has an image such as OACD (Fig. 6) in the state space for the model under consideration. The segment OA describes the sudden application of the strain; the relaxation segment ACD is the path that the state point traverses during the subsequent time period while ϵ is kept constant. Note that both the stress and the state of the material relax to zero during a relaxation test.

A creep test involves a suddenly applied stress which is maintained subsequently. In view of Eqs. (7) and (8), a suddenly applied stress causes a sudden strain which brings the state point, say, to A (Fig. 6). During the subsequent time period, the stress and hence, by Eq. (7), the state variable q_1 are kept constant. We thus have for $\tau > 0$

$$q_1 = \frac{\sigma_0}{c_1}$$

$$q_2 = \frac{\sigma_0}{c_2} [1 - e^{-(c_2/\mu_2)\tau}] \quad (12)$$

and

$$\epsilon(\tau) = \frac{\sigma_0}{c_1} + \frac{\sigma_0}{\mu_3} \tau + \frac{\sigma_0}{c_2} [1 - e^{-(c_2/\mu_2)\tau}]$$

The second equation in (12) is obtained by integration of the last equation in (8). The last equation in (12) then follows from a simple integration of the first equation in (8).

We observe from the above equations that: (a) the state point moves during a creep test ($\tau > 0$) along a line parallel to the q_2 axis; (b) the state point becomes practically stationary at point B for times greater

than μ_2/c_2 ; (c) for $\tau \gg \mu_2/c_2$ the strain increases "linearly" with time and therefore steady creep takes place. We note that at point B, the vector $\vec{g}$ is parallel to the q_1 axis.

As the above remarks show, the material defined by the model of Fig. 5 exhibits full relaxation of stress under constant strain. Conversely, under constant stress, steady creep takes place for long times.

We now consider the modification of this model obtained by the removal of the dashpot with viscosity μ_3 . This can be achieved by letting $\mu_3 \rightarrow \infty$. The resulting model is the familiar three-element model of linear viscoelasticity. Relaxation lines for this model are parallel and straight (Fig. 7). In contrast with the previous model, the relaxation vector $\vec{g}$ is zero not only at the origin but along the line

$$c_1 q_1 - c_2 q_2 = 0 \quad . \quad (13)$$

The images of typical relaxation and creep curves are shown in Fig. 7. The relaxation test now ends at the point D of the zero relaxation line defined by Eq. (13). The stress does not relax completely at the end of a relaxation test.

The creep test has the image OAB in the state space (Fig. 7). At B, as the last equation of (12) shows with $\mu_3 = \infty$, the development of strain stops, so that this material does not exhibit steady creep.

7. MOTIONS IN STATE SPACE IN THE CASE OF A NON-LINEAR CREEPING MATERIAL

The previous section provides one with a starting point in constructing the motions in state space for a non-linear material such as the one discussed in Section 2.

A study of Fig. 1, which summarizes briefly the mechanical behavior of 1100 aluminum, would show that the behavior of the model of Fig. 5 has certain similarities to that of 1100 aluminum. The principal similarity is the presence of steady creep in 1100 aluminum. But there are also many serious dissimilarities. These are the lack of creep at low stresses, the lack of strain recovery upon complete unloading (test 5 in Fig. 1), etc.

Obviously, if one takes the model of Fig. 5 as a starting point in the representation of behavior of aluminum, then one must introduce modifications in Eqs. (7) and (8). One of these modifications may be that the number of state variables must be increased. We leave the discussion of this possibility to the next section.

Now, confining ourselves to two state variables, we discuss the modifications needed in Eqs. (7) and (9), largely in terms of the geometric setting introduced earlier.

We first observe that in tests involving fast straining, 1100 aluminum behaved in an elastic manner.³ This is reminiscent of certain effects observed by Sternglass and Stewart¹¹ in the propagation of small-amplitude waves in a prestressed elastic-plastic solid. These waves propagated with the speed based on the elastic modulus of the material, not with the speed based on the current tangent modulus. These remarks lead us to suggest that we would not modify Eq. (7) and, moreover, in Eq. (9) we would keep $\vec{h}$ as before; namely $\vec{h}$ would have the components 1 and 0; c_1 would then be interpreted as the modulus of elasticity and q_1 as the state variable which measures average lattice strain in this polycrystalline material. Note that an element of the material may deform by means of dislocation motion and intragranular sliding without any change in the average lattice straining.

The modifications are therefore needed only in the vector field $\vec{g}(\vec{q})$. Since the material behaves elastically below a threshold or yield stress, $\vec{g}$ must be zero along a segment on the q_1 axis which contains the origin.

On the other hand, test 5 of Fig. 1 shows that upon unloading, the recovery in strain is only elastic. This suggests that $\vec{g}$ must also be zero along a similar segment on the q_2 axis.

These observations would lead to the assumption that $\vec{g}$ must be zero in a region such as the one shown in Fig. 8.

Since the material exhibits steady creep, the relaxation curves above this region must possess points such as B where $\vec{g}$ is parallel to the q_1 axis. In fact, above the region of no relaxation the shape and distributions of relaxation curves must be rather like those in Fig. 6. However the distribution of the magnitude of the relaxation vector $\vec{g}$ along a relaxation line must be such that the results of tests such as tests 3 and 4

could be accommodated. A little reflection will show that $|\vec{g}|$ must be small in the region limited by the lines $Y_1B_1B_2$ and Y_1Y_2 . Above the line Y_1Y_2 , $\vec{g}$ must increase sharply as one goes along a relaxation line in the direction opposite to the relaxation motion. This would insure that the representation would give rise to high rates of creep observed at the start of a creep test.

In Fig. 8, OAB_2 would be the image of a creep test. Upon sudden unloading during a creep test (cf. test 5 of Fig. 1) the state point would descend to C along a line of fast deformation. If C happens to be below the line of Y_3 and Y_4 , then the state point would move left towards the point Y_3 . This movement comes about because of the fact that $\vec{g}$ at C is as shown in Fig. 8; during the full unloading, $\sigma = 0$ and hence, by Eq. (7), which we retained, $q_1 = 0$. In order to keep $q_1 = 0$, the test specimen must decrease its size, namely ϵ^* must be less than zero, in view of the first equation of (9)

$$q_1^* = g_1 + \epsilon^* = 0$$

and the fact that $g_1 > 0$ at a point such as C. That no appreciable strain rates are observed during full unloading in test 5 is suggestive that the relaxation lines below Y_3Y_4 must be nearly horizontal.

Now consider the reloading part of test 5. This would have the image Y_3D and DB_2 in state space. Along Y_3D the stress would reach its previous value as the strain increases by an amount σ_0/c_1 . Under constant stress the state point would then move from D to B_2 . This would show that during this episode $\epsilon^*(\tau)$ would assume values that it had during a portion of the creep test OAB_2 .

We see that the availability of tests of types 1-5 (Fig. 1) for the material of interest enables us to arrive at certain important conclusions concerning the happenings in state space. We shall discuss in the next section further types of tests which must be conducted in order to answer more detailed questions so that Eqs. (4) and (6') may be set up with more confidence.

8. DO WE NEED MORE THAN TWO STATE VARIABLES?

We now raise the question which constitutes the heading of this section. The answer to this question must be affirmative. In a complicated material such as a dislocation-full polycrystalline solid, a variety of physical phenomena are taking place during deformation processes. It would be unreasonable to assume that these phenomena can be represented by only two numbers.

Here one must point out that the question of number of state variables is intimately connected with the accuracy we want from the mathematical representation. This, in turn, is related to the type of strain or stress histories which are of interest to us. If we are interested only in very slowly varying strain histories, then we may achieve a reasonable accuracy in representation by a few state variables.

With these cautionary remarks, we proceed to look for important signs in the test results of Fig. 1 which indicate the need for more state variables. Tests 3 and 5 are crucial in this respect. If the transient creep observed, after incremental loading (test 3) or after reloading (test 5), is strong, namely if it involves high rates of straining, then the indication would be that we need more state variables. To explain the reasoning behind this conclusion, observe the image of test 5 in Fig. 8. During episode DB_2 , a part of the initial creep curve is repeated according to the model with two state variables. Experimental evidence suggests that this is not so; there is evidence of fresh transient creep after reloading. This sort of transient creep can be achieved only if one is allowed to leave the plane of (q_1, q_2) ; hence the need for one more state variable.

It would be of interest to point out the need for further tests in order to gain more information on the relaxation lines, which are of prime importance in the description of mechanical behavior.

One would need experiments to determine the shape of relaxation lines in the region bounded by $Y_1B_1B_2$ and Y_1Y_2 . These experiments would involve a creep test (such as OAB_2) followed by a relaxation test ($\epsilon = \text{constant}$) which would have the image B_2E_2 . One would also like to learn more about the region below Y_3Y_4 in Fig. 8. This would lead to tests which involve complete unloadings of various durations from various stress levels.

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Fig. 1. Mechanical Behavior of 1100 Aluminum at 300°F.

Fig. 2. Strain Histories for Two Initially Identical Test Specimens, Giving Rise to Different Strains but the Same Stress at Time t .

Fig. 3. Strain Histories (from Fig. 2) Extended from Time t to Time T , Showing that for Small Strains the Extended Portions for the Two Specimens Differ by a Constant.

Fig. 4. Strains Applied in Linear Segments.

Fig. 5. Model of a Viscoelastic Solid.

Fig. 6. Motions in the State Space for Model of Fig. 5.

Fig. 8. State Space of a Nonlinear Material.

AN ELASTIC-PLASTIC CONTINUUM THEORY FOR ARTIFICIAL GRAPHITE*

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(Abstract only)

A small-deformation, elastic-plastic continuum theory for the description of the behavior of artificial graphite in the range from room temperature to a few hundred degrees Fahrenheit is derived. Artificial graphite, in this case, designates a family of materials which are made from petroleum coke and coal-tar pitch and may be either molded or extruded. The applicability of the theory is not restricted to these filler and binder combinations, however. The materials considered exhibit essentially time-independent elastic-plastic behavior; they are anisotropic, and they undergo permanent volume change when tested in simple tension and compression. The continuum description developed is based on generalizations, to combined states of stress, of observations from uniaxial test results, these observations being typical for the entire family considered.

This paper first describes a model of loading surface behavior and gives a qualitative description of surface development which is consistent with loading-unloading-reloading response. In this case, a loading surface does not form the boundary in stress space of a region of purely reversible response, and the loading surface behavior is significantly different from that which forms the basis for the classical plasticity theories.

General plastic constitutive equations are derived to describe the behavior postulated. These are then specialized. The anisotropy and plastic compressibility of graphite are reflected in the formulations, and the resulting equations are used in two examples. In one case, the predictions for a uniaxial specimen are compared with experimental results; in the second, comparisons are made for an internally pressurized, thin-walled cylinder. In each case, the loading and unloading responses are considered.

The research represented by this abstract is reported in detail elsewhere.¹⁻⁴

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✓ STRESS-STRAIN PROPERTIES OF GRADE ATJ GRAPHITE
UNDER COMBINED STRESSES

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ABSTRACT

A generalized stress-strain relation for a transversely isotropic, nonlinear material has been derived by using deformation theory of plasticity. It was assumed that the material anisotropy does not change with deformation and that the degree of nonlinearity is independent of orientation and can be described by a single-valued function of the stress state.

Combined stress tests were conducted on Grade ATJ graphite cylindrical specimens by subjecting them to combinations of internal, external, and axial pressures. In most tests, the stress ratio was maintained constant and the loads were increased monotonically. The longitudinal axis of the cylindrical specimens was always parallel to the symmetry axis of the material. In addition, uniaxial tensile and compressive stress-strain properties in the directions parallel and perpendicular to the material symmetry axis were determined.

The effective stress and effective strain of Grade ATJ graphite were calculated from combined and uniaxial stress test data. Correlation between the effective stress and the effective strain for the combined and uniaxial stress tests was satisfactory. Agreement between the biaxial stress-strain relations measured in the biaxial tests and the biaxial stress-strain relations predicted from the uniaxial stress-strain properties was also found to be satisfactory. The fracture strength of Grade ATJ graphite under combined stresses was compared with various theories of fracture.

INTRODUCTION

In the design of machine and structural components made from graphite, the designer must be able to predict the plastic stress-strain relations and fracture strength under various states of combined stresses from the uniaxial stress-strain properties. Predictions of the stress-strain relations under combined stresses have been made with some degree of success for ductile materials by correlating certain invariant characteristics of the states of stress and strain which are often referred to as the effective stress and effective strain. The relationship between the effective stress

and effective strain is commonly called the generalized or universal plastic stress-strain relation. A number of theories of fracture have been proposed for predicting fracture strength under combined stresses of anisotropic materials. The applicability of these theories of fracture depends on the type of materials and the degree of anisotropy. Furthermore, information on the stress-strain and fracture behavior of brittle materials under combined stresses is limited in both quantity and scope.

Unlike most ductile materials, graphites exhibit limited ductility, moderate anisotropy and nonhomogeneity, nonlinear stress-strain behavior, and change in volume under uniaxial stresses. The applicability of the existing theories of plastic flow and fracture to a class of brittle materials such as graphites has not been demonstrated. In this paper, a generalized stress-strain relation was derived for transversely isotropic materials which have nonlinear stress-strain properties; the stress-strain relations and fracture strength under uniaxial stress and various states of combined stresses were determined experimentally for Grade ATJ graphite, and the experimental results were compared with stress-strain relations under combined stresses predicted by the proposed theory and with biaxial fracture strength predicted by various theories of fracture for anisotropic materials.

A GENERALIZED STRESS-STRAIN RELATION FOR TRANSVERSELY ISOTROPIC MATERIAL EXHIBITING NONLINEAR STRESS-STRAIN PROPERTIES

It is known that the complete plastic behavior of a material is better predicted by the incremental theory of plasticity than the deformation theory of plasticity. However, the two theories of plasticity lead to identical results under the following conditions:^(1,2) (a) the elastic strain is negligible, (b) the loadings are increased monotonically and continuously, and (c) the stress ratio is maintained constant during the loading. It will be shown in the subsequent sections that Grade ATJ graphite exhibits negligible elastic behavior and that most of the combined stress tests in this investigation were conducted under monotonically and continuously increasing loads and at constant stress ratios. Therefore, for these tests, the mathematically simpler deformation theory can be applied.

A generalized form of the nonlinear stress-strain relation for anisotropic materials can be given by Eq. (1):

$$\epsilon_i = \sum_j a_{ij} \sigma_j \phi, \quad (i, j = 1, 2, \dots, 6) \quad (1)$$

where the ϵ_i are the total strain components, σ_j are the stress components, a_{ij} are the constants of anisotropy, and ϕ is the proportionality function. This function is defined by:

$$\phi = \bar{\epsilon} / \bar{\sigma}, \quad (2)$$

where $\bar{\epsilon}$ is the effective strain and $\bar{\sigma}$ is the effective stress which are defined later. It is assumed that the degree and direction of material anisotropy do not change during deformation so that the constants of anisotropy a_{ij} in Eq. (1) are independent of the stress state. It is further assumed that the degree of nonlinearity of the stress-strain behavior is the same for each stress component. Thus, the degree of nonlinearity can be described by a proportionality function ϕ which can be defined uniquely for a given value of the effective stress. This function can be determined from the uniaxial tensile and com-

pressive stress-strain relations and can be used in the prediction of the stress-strain behavior under combined stress loadings.

There are 36 constants of anisotropy in Eq. (1). Because of the symmetry property of the constants of anisotropy $a_{ij} = a_{ji}$ and the assumption that the material has transversely isotropic symmetry, the number of independent a_{ij} is reduced to 5: a_{11} , a_{33} , a_{12} , a_{13} , and a_{44} . A further simplification on Eq. (1) can be made by choosing the directions of the coordinate axes for the stresses to coincide with the directions of the symmetry axes for the material and by considering only stress distributions which are symmetrical with respect to the symmetry axis of the material. Under these conditions, the shear stress components in Eq. (1) vanish. It will be shown in the subsequent section that the foregoing conditions are justified for the present experimental investigations. Therefore, the nonlinear stress-strain relations of interest become

$$\begin{aligned}\epsilon_1 &= (a_{11}\sigma_1 + a_{12}\sigma_2 + a_{13}\sigma_3) \phi, \\ \epsilon_2 &= (a_{12}\sigma_1 + a_{11}\sigma_2 + a_{13}\sigma_3) \phi, \text{ and} \\ \epsilon_3 &= (a_{13}\sigma_1 + a_{13}\sigma_2 + a_{33}\sigma_3) \phi.\end{aligned}\quad (3)$$

The stress components, in terms of the strain components, can be obtained from Eqs. (3) and are given by

$$\begin{aligned}\sigma_1 &= (k_{11}\epsilon_1 + k_{12}\epsilon_2 + k_{13}\epsilon_3)/\phi, \\ \sigma_2 &= (k_{12}\epsilon_1 + k_{11}\epsilon_2 + k_{13}\epsilon_3)/\phi, \text{ and} \\ \sigma_3 &= (k_{13}\epsilon_1 + k_{13}\epsilon_2 + k_{33}\epsilon_3)/\phi,\end{aligned}\quad (4)$$

where

$$\begin{aligned}k_{11} &= (a_{11}a_{33} - a_{13}^2)/\Delta, \\ k_{12} &= (a_{13}^2 - a_{12}a_{33})/\Delta, \\ k_{13} &= (a_{12} - a_{11})a_{13}/\Delta, \\ k_{33} &= (a_{11}^2 - a_{12}^2)/\Delta, \text{ and} \\ \Delta &= (a_{11}^2 - a_{12}^2)a_{33} + 2(a_{12} - a_{11})a_{13}^2.\end{aligned}\quad (5)$$

The product of the effective stress and effective strain is assumed to be given by

$$\bar{\sigma} \bar{\epsilon} = \sigma_1 \epsilon_1 + \sigma_2 \epsilon_2 + \sigma_3 \epsilon_3. \quad (6)$$

The expression for the effective stress for the present case can then be obtained by the substitution of Eqs. (2) and (3) into Eq. (6):

$$\bar{\sigma} = [a_{11}(\sigma_1^2 + \sigma_2^2) + a_{33}\sigma_3^2 + 2a_{12}\sigma_1\sigma_2 + 2a_{13}(\sigma_2\sigma_3 + \sigma_3\sigma_1)]^{\frac{1}{2}}, \quad (7)$$

and the corresponding expression for the effective strain can be derived from Eqs. (2), (4), and (6):

$$\bar{\epsilon} = [k_{11}(\epsilon_1^2 + \epsilon_2^2) + k_{33}\epsilon_3^2 + 2k_{12}\epsilon_1\epsilon_2 + 2k_{13}(\epsilon_2\epsilon_3 + \epsilon_3\epsilon_1)]^{\frac{1}{2}}. \quad (8)$$

EXPERIMENTS

Grade ATJ Graphite

Grade ATJ is a fine grain molded graphite with approximately transversely isotropic properties. For transversely isotropic materials, the properties in the plane perpendicular to the symmetry axis of the material are independent of direction in the plane but are different from those in the direction of the symmetry axis. The symmetry axis of the material approximately coincides with the direction of the molding pressure. Mechanical properties of Grade ATJ graphite will be specified with respect to a Cartesian coordinate system having the X_1 and X_2 axes in the plane of transverse isotropy and the X_3 axis parallel to the symmetry axis of the material.

Uniaxial Stress Tests

Uniaxial tensile and compressive tests were conducted on Grade ATJ graphite specimens having the longitudinal axis oriented parallel or perpendicular to the symmetry axis of the material. Flat coupons with dog-boned gauge section were used in the uniaxial tensile tests, and columns with square cross section were used in the uniaxial compressive tests. All of the uniaxial tensile and compressive test specimens were prepared from the same block of Grade ATJ graphite from which all the cylindrical specimens for combined stress tests were prepared. The longitudinal and transverse strains ϵ_1 , ϵ_2 , and ϵ_3 , were measured with strain gages. The stress-strain curves of Grade ATJ graphite are nonlinear and nonconservative. Uniaxial tensile and compressive stress-strain property data which will be used in the prediction of stress-strain relations and fracture strength under combined stresses are given in Table 1. The values of the elastic compliance constants, S_{ij} , and engineering material constants were determined from the slopes of the stress-strain curves in the limit of zero stress.

Combined Stress Tests

Stress-strain relations and fracture strength under various states of combined stresses have been determined for Grade ATJ graphite by using thin-walled cylindrical specimens subjected to combinations of internal, external, and axial pressures in the pressure chamber shown in Fig. 1. The specimen longitudinal axis was oriented parallel to the symmetry axis of the material, and the test specimens had the following dimensions: inside diameter, 0.885 inch; outside diameter, 0.985 inch; and length, 3.50 inches. For most specimens, the variation of the wall-thickness throughout the test specimens was less than 0.002 inch. A thin layer of synthetic rubber coating was applied to strain gages and specimen surfaces to prevent the hydraulic oil from damaging the strain gages and penetrating into pores when pressure was applied. A thin neoprene bag was inserted inside or placed outside of the test specimens for the same purpose when strain gages were not mounted on the test specimens.

The specimen assembly, consisting of a test specimen and two steel end caps, is shown inside the pressure chamber. The interior surfaces of the steel end caps were slightly tapered, and the steel caps were attached

to the test specimen with epoxy cement. Since the modulus of the epoxy cement is much lower than that of the test specimens, the test specimens were allowed to deform without serious end constraints. Two steel tubes were welded to the upper steel end cap to provide the inlet and outlet ports for internal pressure. The bore of the pressure chamber and the outside surface of the steel end caps were carefully machined to insure that the friction due to "O" ring seals was reduced to a minimum. The strain gage wires were led out from the pressure chamber by means of connectors normally used for thermocouple wires. Four strain gages, two longitudinal and two tangential, were mounted at diametrically opposite locations on the outside surface of most of the test specimens. The corrections for transverse sensitivity of the strain gages were made on the measured strains.

The application of two pressures was usually necessary to obtain a desired state of combined stresses. For most tests, the ratio of the two pressures was maintained constant to provide the loading condition of constant stress ratio. The pressure from the hydraulic power unit could be increased or decreased at prescribed rates by means of a linear actuator which was installed in the pressure relief valve. Pressures were measured by strain-gage type pressure transducers. Pressure-strain relations for the test specimens with strain gages and pressure-time relations for test specimens without strain gages were plotted by X-Y recorders.

The tangential and radial stresses were calculated by Lamé's equations, and the longitudinal stress was calculated by Eq. (9):

$$\sigma_3 = [a^2 P_1 + (d^2 - b^2) P_0 - d^2 P_e] / (b^2 - a^2), \quad (9)$$

where a is the inside radius of test specimen, b is the outside radius of test specimen, d is the inside radius of the pressure test chamber, P_1 is the internal pressure, P_0 is the external pressure, and P_e is the axial end pressure. The radial stress, which is equal to the oil pressure at the surface of the test specimen, was usually small compared with the tangential and longitudinal stresses.

The pressure-longitudinal strain relation and the pressure-tangential strain relation for a cylindrical specimen subjected to internal pressure are shown in Fig. 2. The nonlinear stress-strain behavior and the nonconservative response under cyclic internal pressure loading shown in Fig. 2 are similar to those obtained under uniaxial stress loading.

COMPARISON OF THEORY WITH EXPERIMENTAL RESULTS

Generalized Stress-Strain Relation

In this study, the value of each constant of anisotropy a_{ij} is selected to be a dimensionless quantity having 10^6 times the value of the corresponding elastic compliance constant S_{ij} given in Table 1; the proportionality function ϕ then has the value of 10^{-6} in./lb in the limit of zero stress.

The effective stress and effective strain for the uniaxial stress tests can be calculated by using Eqs. (7) and (8) and the measured values of applied stress and corresponding three strain components. The value of ϕ as a function of $\bar{\sigma}$ was computed from the average effective stress-effective strain curve for all the uniaxial tensile and compressive tests. The radial strain, ϵ_2 , in the combined stress test cylinders was not measured. The magnitude of ϵ_2 is usually small in comparison to the magnitudes of ϵ_1 and ϵ_3 , and the approximate value of ϵ_2 can be computed from the second expression of

Eqs. (3) together with the value of ϕ determined from the average effective stress-effective strain curve for all the uniaxial stress tests. The effective stress-effective strain relations for the combined stress tests were obtained from the values of $\bar{\sigma}$ calculated with Eq. (7) and the corresponding values of $\bar{\epsilon}$ computed with Eq. (8) by using the calculated values of ϵ_2 and the experimental values of ϵ_1 and ϵ_3 .

The effective stress-effective strain relations for the uniaxial stress tests and the ten combined stress tests which were conducted under the condition of constant stress ratio are shown in Fig. 3. The average effective stress-effective strain curve for the uniaxial stress tests is shown by the solid curve. The correlation between the effective stress and the effective strain is quite satisfactory for both uniaxial and biaxial stress tests. In fact, the scatter of the effective stress-effective strain relations for the combined stress tests is not greater than that for the uniaxial stress tests; consequently, the stress-strain relations under combined stresses for Grade ATJ graphite can be predicted with the same degree of accuracy which is obtainable in the determination of the uniaxial stress-strain relations.

Stress-Strain Relations Under Combined Stresses

In the foregoing section, it was shown that the generalized stress-strain curve for Grade ATJ graphite exists and can be determined from the uniaxial stress-strain properties. In this section, it will be shown that stress-strain relations under combined stresses can be predicted satisfactorily from the data obtained from the uniaxial stress tests: elastic compliance constants, S_{ij} , and the proportionality function, ϕ . Stress-strain relations in the tangential and longitudinal directions for the combined stress test specimens were calculated by means of the first and third expressions of Eqs. (3). The experimental and calculated stress-strain relations in the tangential and longitudinal directions for the specimens tested in four different biaxial stress quadrants are shown in Figs. 4 to 7. The value in parentheses in Figs. 4 to 7 denotes the stress ratio at which the specimen was tested. The tangential and longitudinal stresses are denoted, respectively, by σ_1 and σ_3 . The negative sign indicates that the stress was in compression. The experimental stress-strain curves are shown by solid curves, and the calculated stress-strain curves are shown by dashed curves.

The stress-strain relations in the tangential and longitudinal directions for a specimen tested under internal pressure are shown in Fig. 4. The stress ratio was approximately 0.5; the tangential and longitudinal stresses were both tensile. In this case, the theory somewhat underestimated the value of the longitudinal strain. The stress-strain relations in the tangential and longitudinal directions for a specimen subjected to external pressure at the stress ratio of 0.43 are shown in Fig. 5. In this case, the external pressure also exerted axial tension on the specimen; therefore, the tangential stress was compressive, and the longitudinal stress was tensile. The stress-strain relations in the tangential and longitudinal directions for a specimen tested under a simultaneous application of axial and external pressures at the stress ratio of 0.66 are shown in Fig. 6. In this case, the tangential and longitudinal stresses were both compressive. The stress-strain relations in the tangential and longitudinal directions for a specimen tested under a combination of internal and axial pressures are shown in Fig. 7. In this case, the tangential stress was in tension, and

the longitudinal stress was in compression; the ratio of the stresses for this specimen was 1.05. Agreement between the experimental and calculated biaxial stress-strain relations for the ten cylindrical specimens was considered quite satisfactory for most engineering applications.

Fracture Strength Under Biaxial Stresses

Since, in the present investigation, the magnitude of the radial stress is much smaller than the magnitudes of the tangential and longitudinal stresses, it is necessary to consider the fracture strength only under the states of biaxial stresses: tangential and longitudinal stresses. Most of the theories of fracture for anisotropic materials predict the fracture strength envelope under the states of biaxial stresses to be in the form of either a single ellipse or a discontinuous curve composed of four separate fracture envelopes which are consistent with the failure mode operative in each quadrant of biaxial stresses. The separate fracture envelopes are segments of four different ellipses. Thus, these theories of fracture are expressed in the form of the quadratic equation for an ellipse in terms of two principal stresses. Constants in the quadratic equations are determined from the values of the uniaxial fracture strengths.

Four theoretical fracture strength envelopes are compared with the measured fracture strength in Fig. 8. Each circle represents the fracture strength for an individual specimen. These fracture strength envelopes, in the form of a continuous ellipse, were proposed by Gol'denblat and Kopnov⁽³⁾, Hoffman⁽⁴⁾, Stassi-D'Alia⁽⁵⁾, and Hill⁽¹⁾. Figure 8 shows that the theories of fracture proposed in References 3, 4, and 5 are in good-to-fair agreement with the experimental data in the biaxial-tensile and tensile-compressive stress quadrants. The fracture strength envelope calculated by Hoffman's theory appears to give the best prediction of the experimental data. Fracture strength envelopes predicted by the theories of fracture proposed by Norris⁽⁶⁾ and Ely⁽⁷⁾ are compared with the measured fracture strength in Fig. 9. These fracture strength envelopes are composed of four separate curves and are in fair agreement with the experimental data only in the tensile-compressive stress states.

Both Figs. (8) and (9) show that the measured fracture strength for Grade ATJ graphite in the biaxial-compressive stress state was considerably lower than the values predicted by all of the theories of fracture except Hill's theory. This discrepancy was believed to be, in part, due to the experimental difficulties encountered in the determination of fracture strength of Grade ATJ graphite under the state of biaxial compressive stresses. Several cylindrical specimens with gauge length-to-mean diameter ratios (L/D) of 1.23, 1.87, 2.67, and 3.64 were tested in biaxial compression at the stress ratio of approximately 0.78. In all cases, the diameters of the cylindrical specimens were identical to those of the regular cylindrical specimens. The gauge length-to-mean diameter ratio for the regular cylindrical specimens was 2.67. The values of fracture strength for specimens with different L/D ratios are shown in Fig. 9. The fracture strength increased almost 60 percent as the L/D ratio decreased from 3.64 to 1.23; nevertheless, the fracture strength for the specimens with an L/D ratio of 1.23 was still low in comparison with the values predicted by the theories. No apparent change in fracture strength occurred when the L/D ratio was decreased from 2.67 to 1.87. Photographs of the fractured specimens are shown in Fig. 10. The photographs revealed that all the cylindrical specimens fractured in the

same mode, regardless of their gauge lengths. Fracture of these specimens was not caused by gross buckling but, rather, was caused by highly localized collapse or buckling of the specimen wall, presumably initiated from an inherent defect in the material. Thus, the fracture strength of Grade ATJ graphite under the state of biaxial compressive stresses apparently cannot be measured accurately with thin-walled cylindrical specimens.

A preliminary investigation was conducted on triaxial compressive tests of Grade ATJ graphite. Two dog-boned solid cylindrical specimens with diameter of 0.500 inch and gauge length of 0.785 inch were tested under triaxial compressive stresses by subjecting them to a simultaneous application of external and axial end pressures. The specimen longitudinal axis was oriented parallel to the symmetry axis of the material. The stress state at fracture for these specimens was approximately 13,000 psi for the longitudinal stress, σ_3 , and approximately 2,200 psi for the radial stresses, $\sigma_1 = \sigma_2$. As expected, the preliminary results indicate that the uniaxial compressive strength of Grade ATJ graphite increases if a radial hydrostatic pressure is applied to the specimen during the test.

CONCLUSIONS

A generalized stress-strain relation for a transversely isotropic, non-linear material has been proposed by using deformation theory of plasticity without assuming that the volume remains constant. Combined and uniaxial stress tests were conducted on Grade ATJ graphite. Comparison of the theory and the experimental results showed that the effective stress and the effective strain for Grade ATJ graphite could be correlated satisfactorily for both uniaxial and combined stress tests and also that the stress-strain behavior under combined stresses for Grade ATJ graphite could be predicted with reasonable accuracy from the uniaxial stress test data. Comparison of the appropriate theories of fracture for anisotropic materials with the measured fracture strength indicated that fracture strength of Grade ATJ graphite under the biaxial-tensile and tensile-compressive stress states could be predicted fairly adequately by some of the theories of fracture.

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Table 1. Mechanical Properties of Grade ATJ
Graphite in Tension and Compression

Elastic Compliance Constants ($10^{-6}\text{in.}^2/\text{lb}$)			
S_{11}	S_{33}	S_{12}	S_{13}
0.61	0.89	-0.06	-0.10

Engineering Material Constants				
Young's Modulus (10^6 psi)		Poisson's Ratio		
$1/S_{11}$	$1/S_{33}$	$-S_{12}/S_{11}$	$-S_{13}/S_{11}$	$-S_{13}/S_{33}$
1.65	1.12	0.10	0.16	0.11

Fracture Strengths (psi)			
Tensile		Compressive	
σ_{t1}	σ_{t3}	σ_{c1}	σ_{c3}
3800	3000	9600	9200

Fig. 1. Pressure Chamber Assembly for Combined Stress Tests.

Fig. 2. Pressure vs Longitudinal and Outside Tangential Strain Curves for Grade ATJ Graphite Cylinder Subjected to Internal Pressure.

Fig. 3. Effective Stress-Effective Strain Relation for Grade ATJ Graphite.

Fig. 4. Biaxial Stress-Strain Relations for Grade ATJ Graphite Cylinder Tested in the Biaxial-Tensile Stress Quadrant.

Fig. 5. Biaxial Stress-Strain Relations for Grade ATJ Graphite Cylinder Tested in the Compressive-Tensile Stress Quadrant.

Fig. 6. Biaxial Stress-Strain Relations for Grade ATJ Graphite Cylinder Tested in the Biaxial-Compressive Stress Quadrant.

Fig. 7. Biaxial Stress-Strain Relations for Grade ATJ Graphite Cylinder Tested in the Tensile-Compressive Stress Quadrant.

Fig. 8. Biaxial Fracture Strength for Grade ATJ Graphite.

Fig. 9. Biaxial Fracture Strength for Grade ATJ Graphite.

Fig. 10. Fractured Cylindrical Specimens with Various Gauge Length-to-Mean Diameter Ratios (Stress Ratio: $-\sigma_3/-\sigma_1 = 0.78$).

THERMOMECHANICS OF RATE-INDEPENDENT MATERIALS WITH MEMORY

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ABSTRACT

A theory previously developed by the author (Ninth Carbon Conference, 1969, and Fifth Southeastern Conference on Theoretical and Applied Mechanics, 1970) for the rate-independent isothermal mechanical response of transversely isotropic materials is thought to be applicable to the room-temperature behavior of certain graphites and metals exhibiting pronounced nonlinear characteristics even at low strain levels.

In this paper, starting with the principles of continuum thermodynamics, a thermomechanical theory for rate-independent response is developed. Heat and temperature effects, as well as mechanical stress and strain, are taken into consideration as prime factors in the theory.

The development begins by postulating that the stress tensor, heat flux vector, and Helmholtz free energy are functionals of the history of the strain tensor, temperature, and temperature gradient vector. Then, by assuming that the free energy functional is rate-independent and possesses an instantaneous modulus, we show by the second law of thermodynamics that (i) the stress and internal energy are independent of the history of the temperature gradient but the heat flux, in general, is not; and that (ii) the stress is derivable from the free energy. The remainder of the paper is devoted to developing a rate-independent representation of the thermomechanical response for isotropic materials with nonlinear characteristics and permanent memory. Primary consideration is given to the constitutive equations for the stress and heat flux.

This paper arose out of some experimental work which we did some years ago for Los Alamos Scientific Laboratory. Initially we were concerned with the mechanical response, then with the thermal response, and now with the thermomechanical coupled response of nonlinear inelastic materials such as graphites.

The type of mechanical response which motivated our work is shown in Fig. 1, which illustrates the highly nonlinear memory-dependent response of typical graphites. We attempt here to lay down a thermomechanical memory-dependent nonlinear theory based upon the basic principles of continuum mechanics.

A thermomechanical process is defined by the eight functions given in Eqs. (1):

(i) Spatial position	$\underline{x} = \underline{x}(x, \tau)$	
(ii) Symmetric stress tensor	$\underline{T} = \underline{T}(x, \tau)$	
(iii) Specific body force	$\underline{b} = \underline{b}(x, \tau)$	
(iv) Specific internal energy	$e = e(x, \tau)$	
(v) Heat flux vector	$\underline{q} = \underline{q}(x, \tau)$	(1)
(vi) Rate of heat supply	$r = r(x, \tau)$	
(vii) Specific entropy	$\eta = \eta(x, \tau)$	
(viii) Absolute temperature	$\theta = \theta(x, \tau)$	

where

x is a material point,

τ is time, $-\infty < \tau < \infty$.

We make no attempt here to give a physical interpretation of these functions, although most of them have a fairly obvious physical meaning.

These eight functions of time and material points must satisfy the field equations, Eqs. (2), resulting from the balance laws of momentum and energy:

Momentum

$$\rho \ddot{\underline{x}} = \text{div } \underline{T} + \rho \underline{b}$$

Energy

(2)

$$\rho \dot{e} = \text{tr } \underline{T} \text{ grad } \dot{\underline{x}} - \text{div } \underline{q} + \rho r$$

We find it convenient to introduce the deformation gradient, $\underline{F}$, the referential stress, $\underline{S}$, and the referential heat flux vector, $\underline{Q}$, as defined in Eqs. (3):

$$\begin{aligned}\underline{F} &= \text{grad } \underline{x} \\ \underline{S} &= (\det \underline{F}) \underline{T} (\underline{F}^T)^{-1} \\ \underline{Q} &= (\det \underline{F}) \underline{F}^{-1} \underline{q}\end{aligned}\tag{3}$$

We can then write the balance laws in terms of these newly defined quantities. These referential field equations are

$$\begin{aligned}\rho_o \ddot{\underline{x}} &= \text{div } \underline{S} + \rho_o \underline{b} \\ \rho_o \dot{e} &= \text{tr } \underline{S}^T \dot{\underline{F}} - \text{div } \underline{Q} + \rho_o r\end{aligned}\tag{4}$$

To the balance laws of momentum and energy, we now adjoin a dissipation postulate, or, if you prefer, a second law of thermodynamics. We take the Clausius-Duhem inequality, and the resulting field inequality is the dissipation postulate shown in Eq. (5) or Eq. (6).

$$\rho_o \dot{\eta} + \text{div } \frac{\underline{Q}}{\theta} - \frac{\rho_o r}{\theta} \geq 0\tag{5}$$

or

$$\rho_o \theta \dot{\eta} - \rho_o \dot{e} + \text{tr } \underline{S}^T \dot{\underline{F}} - \frac{1}{\theta} \underline{Q} \cdot \underline{h} \geq 0\tag{6}$$

$$\underline{h} = \text{grad } \theta$$

For convenience, we introduce the specific free energy, ψ , defined in Eq. (7).

$$\begin{aligned}\psi &= \psi(x, \tau) \\ &= e - \theta \eta\end{aligned}\tag{7}$$

Then we can rewrite our dissipation inequality in terms of the free energy and the referential quantities. This inequality is given by

$$\rho_o \dot{\psi} + \rho_o \dot{\theta} \eta - \text{tr} \underline{\underline{S}} \underline{\underline{F}}^T + \frac{1}{\theta} \underline{\underline{Q}} \cdot \underline{\underline{h}} \leq 0 \quad (8)$$

As we said at the beginning, we wish our final formulation to be a constitutive equation which depends upon the history of the thermo-mechanical process. To this end we define the history function f^t of the function f by the following procedure:

Let f be some function of time τ for

$$-\infty < \tau \leq t$$

where

t is the present time.

Define the history of f by f^t

$$f^t(s) = f(t-s) \quad 0 \leq s < \infty$$

with

$$f^t(0) = f(t)$$

In this manner we can define the histories of the deformation gradient, the temperature, and the temperature gradient:

Deformation gradient history	$\underline{\underline{F}}^t$
Temperature history	θ^t
Temperature gradient history	$\underline{\underline{h}}^t$

We are now ready to lay down our general constitutive assumption. We postulate that the stress, internal energy, entropy, heat flux, and free energy are smooth functionals of the history of the deformation gradient, temperature, and temperature gradient, as indicated in Eqs. (9).

$$\left. \begin{aligned} \underline{S}(t) &= \underline{S}(\underline{F}^t, \theta^t, \underline{h}^t) \\ e(t) &= E(\underline{F}^t, \theta^t, \underline{h}^t) \\ \eta(t) &= N(\underline{F}^t, \theta^t, \underline{h}^t) \\ \underline{Q}(t) &= \underline{Q}(\underline{F}^t, \theta^t, \underline{h}^t) \\ \psi(t) &= P(\underline{F}^t, \theta^t, \underline{h}^t) \end{aligned} \right\} \text{All } t \quad (9)$$

These functionals are to describe the thermomechanical response of the material and therefore must satisfy the balance laws of momentum and energy and also the dissipation inequality. Their being functionals of histories rather than functions clearly incorporates the memory aspects of the response.

We now turn to the question of rate-independence. By rate-independence we mean that a change in the time scale of any experiment would not change the observed response. Formally, if we rescale the time parameter, the response functionals will not be affected by any such rescaling:

Let

$$\varphi = \varphi(s)$$

be a time-rescaling function.

Definition: A functional $P(\underline{f}^t)$ is rate-independent if

$$P(\underline{f}^t) = P(\underline{f}^t \cdot \varphi)$$

for all rescaling function φ .

For such rate-independent functionals, Theorem 1 states that the instantaneous rate of change of the response is linearly proportional to the instantaneous rate of change of the inputs. The proportionality factor is called the instantaneous modulus and is dependent upon the previous history of the inputs.

Theorem 1. If $P(\dot{f}^t)$ is rate-independent, then

$$\dot{P}(\dot{f}^t) = -M_{\dot{f}}^t \cdot \dot{f}^t$$

where $M_{\dot{f}}^t$ is the instantaneous modulus of P .

The fundamental result of this paper is contained in Theorem 2. Essentially, the theorem says that if the free energy P is rate-independent, then the stress, entropy, and internal energy are all rate-independent and all of these are independent of the temperature gradient history. Furthermore, the heat flux vector satisfies the classical heat conduction inequality.

Theorem 2. If $P(\underline{F}^t, \theta^t, \underline{h}^t)$ is rate-independent and possesses an instantaneous modulus, then

- (i) $\underline{S}$, e , η , and ψ are rate-independent,
- (ii) $\underline{S}$, e , η , and ψ are independent of $\underline{h}^t$,
- (iii) $\underline{S}$ and η are derivable from P via

$$\underline{S} = -\rho_0 \frac{M_{\underline{F}}^t}{T}, \quad \eta = M_{\theta}^t$$

- Remark: (i), (ii), and (iii) do not apply to $\underline{Q}$.
 (iv) The heat flux satisfies

$$\underline{Q} \cdot \underline{h} \leq 0$$

Many other classical-like results follow from this theorem. Further observations that can be made here are:

1. Equilibrium heat flux vanishes

$$\underline{Q}(\underline{F}_0^t, \theta_0^t, \underline{0}^t) = \underline{0}$$

2. Gibbs' relation

$$\rho_0 \dot{e} = \text{tr } \underline{S}^T \dot{\underline{F}} + \theta \dot{\eta} \rho_0$$

3. Stress work-isothermal process

$$\int_{t_1}^{t_2} \text{tr } \underline{\underline{S}}^T \dot{\underline{\underline{F}}} dt = \psi(t_2) - \psi(t_1)$$

4. Stress work-isentropic process

$$\int_{t_1}^{t_2} \text{tr } \underline{\underline{S}}^T \dot{\underline{\underline{F}}} dt = e(t_2) - e(t_1)$$

We are now ready to develop specific forms for our constitutive functionals. In doing so we must incorporate whatever symmetry the material may possess in regard to both its mechanical and its thermal properties. In particular, for isotropic materials the results are given in the following equations, in which we introduce the Green strain tensor.

Material symmetry and frame indifference imply that for isotropic materials

$$\underline{\underline{S}} = \underline{\underline{F}} \hat{\underline{\underline{S}}} (\underline{\underline{E}}^t, \theta^t)$$

$$\hat{\underline{\underline{S}}} \text{ is symmetric}$$

$$e = E(\underline{\underline{E}}^t, \theta^t)$$

$$\underline{\underline{Q}} = (\underline{\underline{E}}^t, \theta^t, \underline{\underline{h}}^t)$$

where

$$\underline{\underline{E}} = \underline{\underline{F}}^T \underline{\underline{F}} - \underline{\underline{I}} \text{ is Green strain}$$

and

$$\hat{\underline{\underline{S}}}, E, \text{ and } \underline{\underline{Q}} \text{ are isotropic.}$$

In order to construct rate-independent functionals, we can introduce the arc-length parameter, $\alpha(\tau)$.

$$\alpha(\tau) = \int_0^\tau \sqrt{\text{tr } \dot{\underline{E}}(s) \dot{\underline{E}}(s) + \dot{\theta}(s) \dot{\theta}(s)} \, ds$$

with

$$\alpha(0) = 0, \quad \alpha(t) = L$$

Essentially, $\alpha(\tau)$ is a time-independent parameter which increases monotonically with changes in the strain and temperature of the material. Using this parameter instead of the usual wall clock time parameter, the response of the material becomes dependent upon its internal strain and temperature "clock" rather than on the wall clock. Next, construct the arc-length parameterizations by

$$\bar{\underline{E}}(\ell) = \underline{E} \cdot \alpha^{\dot{}}(\ell), \quad \bar{\theta}(\ell) = \theta \cdot \alpha^{\dot{}}(\ell)$$

where $\alpha^{\dot{}}(\ell)$ is the right inverse of $\alpha(\tau)$.

If we now rewrite the strain and temperature histories in terms of the arc-length parameter, we can easily construct rate-independent functionals. In the example below, we have written a rate-independent isotropic tensor-valued functional which is very similar in form to the type used to describe a linear thermoviscoelastic material by means of single integrals.

Any functional P of the form

$$P(\underline{E}^{\dot{}}, \theta^{\dot{}}) = \bar{P}(\bar{\underline{E}}, \bar{\theta})$$

is rate-independent.

Example:

$$\begin{aligned} \hat{\underline{S}}(\bar{\underline{E}}, \bar{\theta}) &= \int_0^L 2\mu(L-\ell) \dot{\underline{E}}(\ell) \, d\ell \\ &+ \underline{\underline{I}} \int_0^L \lambda(L-\ell) \text{tr } \dot{\underline{E}}(\ell) \, d\ell \\ &+ \underline{\underline{I}} \int_0^L \beta(L-\ell) \dot{\bar{\theta}}(\ell) \, d\ell \end{aligned}$$

is a rate-independent isotropic stress functional.

We observe that if the material functions μ , λ , and β are constants, we recover the theory of linear finite thermoelasticity. On the other hand, thermoviscoelasticity is not included within this representation because of the explicit absence of time. Viscoelasticity in an arc-length sense is included, however. Note that although the integrals appear to be linear in the strain and temperature, they are in actuality nonlinear because of the dependence of the arc-length parameter upon the strain and temperature histories.

In conclusion, we have attempted to use this type of theory in describing the room-temperature mechanical response of various graphites. Figure 2 shows an example of the type of results we have obtained.

I make no claims as to the ultimate usefulness of this theory. This question lies in the hands of you who must attempt to use such theories in your design applications.

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Fig. 1. Cyclic Stress-Strain.

Fig. 2. Load-Strain in 2 or 3 Direction.

POSSIBILITIES FOR APPLICATION OF HIGHER-
ORDER CONTINUUM THEORIES

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ABSTRACT

This paper is essentially a progress report on an investigation being conducted in the Department of Mechanical Engineering at the University of Colorado on the feasibility of applying higher-order continuum theories in predicting the behavior of elementary structures of carbon and carbon composites. Hopefully, the results of this study will establish the credibility of the use of such a theory and predicate the formulation of suitable approximate theories for the analysis of elementary structures.

As an illustration of a continuum theory which takes account of microstructure effects, the field equations of the linear isotropic micropolar elasticity theory are discussed in a summary manner. The essence of the paper is a discussion of the feasibility of and limitations on an experimental study which will result in an indication of the quantitative significance of the inclusion of microstructure effects in the prediction of both material and structural behavior.

As has been repeatedly pointed out by numerous authors, the physical character of carbon composites is such that the utilization of the usual non-polar continuum theories in predicting material behavior is subject to both conceptual and practical limitations. The present paper is in the nature of a progress report on an investigation dealing with the feasibility of the utilization of higher-order continuum theories in predicting such material behavior. Specifically, the present phase of this investigation is concerned with the development of experimental procedures suitable for establishing the credibility of such theories in accounting for secondary or microstructure effects.

During the past decade considerable attention has been given to the development and application of higher-order continuum theories with a resulting

substantial (and perhaps formidable) body of literature readily available. Although a compendium of this literature is obviously well beyond the scope of the present discussion, it is pertinent to recall the significance in this development of the director theory [1], the couple stress theory [2], the multipolar theory [3], and the micropolar theory [4].

As a point of departure for the present discussion, let us briefly review some of the elements of the linear micropolar elasticity theory: The essence of the kinematical considerations of the micropolar theory resides in the deformation of a "small" volume ΔV with center of mass located by position vector $\underline{X}$ into a volume Δv with center of mass located by position vector $\underline{x}$ (Fig. 1). The volume ΔV is assumed to be comprised of n microelements of volume $\Delta V^{(\alpha)}$ of density $\rho^{(\alpha)}$ and with position vector $\underline{Z}^{(\alpha)}$ with respect to the center of mass of ΔV . Thus the mass Δm of ΔV is given by

$$\Delta m = \sum_{\alpha} \rho^{(\alpha)} \Delta V^{(\alpha)} \quad (1)$$

On deformation $\underline{Z}^{(\alpha)}$ is deformed to $\underline{z}^{(\alpha)}$, and in view of the restriction of our discussion to a linear theory we may write

$$\underline{z}^{(\alpha)} = \underline{Z}^{(\alpha)} + \underline{\phi} \times \underline{Z}^{(\alpha)} \quad (2)$$

where the vector $\underline{\phi}$ characterizes the rigid microrotations of the microelements about the center of mass of the volume ΔV .

It is then clear that

$$\underline{x}^{(\alpha)} = \underline{X} + \underline{Z} + \underline{u} + \underline{\phi} \times \underline{Z} \quad (3)$$

where $\underline{u}$ is the displacement vector given by $\underline{x} - \underline{X}$. It follows that

$$d\underline{x}^{(\alpha)} = d\underline{X} + d\underline{Z} + \underline{u}_{,I} dX_I + \underline{\phi} \times d\underline{Z} + \underline{\phi}_{,I} \times \underline{Z} dX_I \quad (4)$$

We then write

$$\underline{u}_{,I} dX_I = E_{IJ} dX_I \underline{E}_J + \underline{R} \times d\underline{X} \quad (5)$$

where E_{IJ} are the Lagrangian strain components, $\underline{R}$ is the rotation vector, and $\underline{E}_J$ are the base vectors of a material Cartesian reference frame. We also write

$$\underline{\phi}_{,I} \times \underline{Z} dX_I = \Gamma_{IJK} Z_J dX_K \underline{E}_I \quad (6)$$

where the third-order tensor

$$\Gamma_{IJK} = e_{ILJ} \phi_{L,K} \quad (7)$$

and where e_{IJK} is the permutational symbol. We define

$$\Gamma_{IJ} = \Gamma_{IJK} Z_K = \Gamma_{(IJ)} + \Gamma_{[IJ]} \quad (8)$$

The dual vector of $\Gamma_{[IJ]}$ is

$$\Gamma_I = \frac{1}{2} e_{IJK} \Gamma_{KJ} \quad (9)$$

Thus

$$d\underline{x}^{(\alpha)} = d\underline{X} + d\underline{Z} + (\underline{R} + \underline{\Gamma}) \times d\underline{X} + \underline{\phi} \times d\underline{Z} + (E_{IJ} + \Gamma_{(IJ)}) dX_I \underline{E}_J \quad (10)$$

It is now clear that the deformation is characterized by micropolar strains and rotations and minirotations in addition to the usual strains and rotations which we now call macrostrain and macrorotation.

Referring now to a spatial coordinate system and using lower-case letters to indicate the spatial representation of the quantities previously discussed, we have, after appropriate linearization:

macrostrain and macrorotation

$$e_{ij} = u_{(i,j)} \quad r_i = \frac{1}{2} e_{ijk} u_{[i,j]}$$

micropolar strain

$$\epsilon_{ij} = e_{ij} + e_{ijk} (r_k - \phi_k) \quad \gamma_{ijk} = e_{ijl} \phi_{l,k} \quad (11)$$

minirotation

$$\gamma_i = \frac{1}{2} e_{ijk} \gamma_{kj} \quad \gamma_{ij} = e_{ijl} \phi_{l,k} \zeta_k$$

We also note that these quantities must satisfy the compatibility conditions

$$\epsilon_{ik,j} - \epsilon_{jk,i} + \gamma_{ikj} - \gamma_{jki} = 0 \quad (12)$$

$$e_{mni} (\gamma_{mnj,k} - \gamma_{mnk,j}) = 0$$

The stress vector T_i and stress couple vector m_i are defined in the usual way, and are related to the stress tensor T_{ij} and stress couple tensor m_{ij} by

$$T_i = T_{ji} n_j \quad m_i = m_{ji} n_j \quad (13)$$

where n_j are the components of a unit vector normal to the surface on which these vectors act. The equilibrium conditions are readily shown to be

$$\begin{aligned} T_{ji,j} + \rho f_i &= 0 \\ m_{ji,j} + e_{ijk} T_{jk} + \rho \ell_i &= 0 \end{aligned} \quad (14)$$

where f_i and ℓ_i are, respectively, the body force and body couple fields.

The constitutive relations of a linear elastic theory are written

$$T_{ij} = A_{ijkl} \epsilon_{kl} \quad m_{ij} = B_{ijkl} \phi_{k,l} \quad (15)$$

where the fourth-order tensors A_{ijkl} and B_{ijkl} are constants. With the assumption of isotropy these relations may be written in the form

$$\begin{aligned} T_{ij} &= \lambda \epsilon_{kk} \delta_{ij} + (\mu + \kappa) \epsilon_{ij} + \mu \epsilon_{ji} \\ m_{ij} &= \alpha \phi_{k,k} \delta_{ij} + \beta \phi_{i,j} + \gamma \phi_{j,i} \end{aligned} \quad (16)$$

Thus a total of six elastic constants is necessary for the material characterization, even in the case of a linear isotropic theory.

It may be shown that the surface boundary conditions consistent with the foregoing field equations are

- (i) either $\underline{u}$ specified or $\underline{T}$ specified
- (ii) either $\underline{\phi}$ specified or $\underline{m}$ specified

We now turn our attention to the objectives of our investigation. We emphasize that we are not confining our attention to the linear micropolar elasticity theory, but have included the summarization of that theory only as a vehicle for discussion.

Ideally, we would wish to experimentally determine the material constants (the six elastic constants in the case of the micropolar theory) and then proceed to the establishment of the credibility of the theory through the experimental verification of the predictions of the theory as regards the behavior of elementary structures. Obviously, the utilization of a material in which microstructure effects are significant is essential, and we must proceed under the hypothesis that a carbon composite is such a material. Indeed, the establishment of the validity (or invalidity) of this hypothesis is a fundamental part of our investigation.

In a conceptual idealization of an appropriate experimental procedure we might envision a filter, a transducer, and an amplifier: the filter to exclude the macrostructure effects and the transducer and amplifier to precisely measure the microstructure effects. Certain observations can be made from a consideration of the field equations of a higher-order theory, e.g. the linear micropolar theory. The microstructure effects (if significant at all) would be significant in the presence of an applied surface couple field which field advantageously possesses a large directional derivative in some direction along the surface. The production of such a field in a laboratory is clearly a most difficult (if not impossible) task. Alternatively, in view of the second of Eqs. (14), a surface stress field with a large directional derivative (and no surface couple field) could produce a substantial stress couple in a region near the region of the load application. The most obvious device for the production of such a surface field is the knife-edge loading. However a knife-edge loading also produces very large displacement gradients in the vicinity

of the load application, and for this reason it provides a significant macrostructure effect on which the superposition of the microstructure effect may be relatively small. We then seek a surface stress field which produces large directional derivatives in some region on the surface but simultaneously provides small displacement gradients in the vicinity of that region.

An experimental device which meets these criteria is achieved through a system of two beams shown symbolically in Fig. 2. For simplicity the beams may be identical in both shape and material composition. Initially the beams are separated by a small gap and two corresponding ends are constrained from deflection and loaded as shown. For sufficiently large intensity of the applied couples, a separation of the two beams will be produced at the other ends. The explanation of this behavior of the system on the basis of elementary (macrostructure) beam theory is not possible. In terms of macrostructure analysis, a more sophisticated beam theory will predict the separation of the ends of the beam and the distribution of the transmitted load in the region of contact as being a surface load with extremely large directional derivative near the edges of the region of contact, particularly in cases where the beams are "thin". Under the assumption that a microstructure beam theory analysis will predict a similar character in the surface traction transmitted throughout the region of contact, we note that microstructure effects will be pronounced at the edges of the contact region and will be evident in the magnitudes of the separation at the ends of the beams. We note that the beam region between the free end and the region of contact is under no load and thus the magnitude of the separation at the end of the beam is linearly proportional to the length of this region. In this manner the experiment itself provides us with our ideally sought "filter, transducer, and amplifier." The microstructure effects dominate the beam behavior in the region immediately adjacent to the free portion; the rigid body rotation of this free portion is a measure of the significance of these effects, and this measure can be amplified by merely choosing a geometric configuration in which this load-free portion will be "long."

A second experimental device which offers promise of providing a similar but more general surface load with the required locally large directional derivative is provided by two overlapping and bonded plates. The linear elastic

generalized plane stress solution of this problem has been treated extensively in the literature and is summarized in detail by Sternberg [5]. For our purposes the significant part of this analysis is the indication that in the region of adhesion the transmitted shearing surface tractions are divided into two parts: a shearing traction distributed throughout the interior of this region, and a line load, called the edge bond traction, transmitted at the edge of the region of adhesion. Clearly a discontinuity in the surface traction accomplished in this manner will be accompanied by an absence of substantial macro-displacement gradients. In a manner similar to that of the experimental procedure involving the two beams, a measurable relative displacement of the two plates (under edge loads) will include the significant microstructure effect produced in the region of surface traction discontinuity.

Obviously, before either of these proposed experiments can be implemented it will be necessary for us to extend the existing approximate theory analysis to include microstructure effects. A number of authors have proposed beam and plate theories which include such effects and are predicated on suitable higher-order continuum theories. Unfortunately the application of such theories to the analysis of either the beam or the plate experiment is not particularly straight-forward and at the present time involves a number of rather subtle questions which we are in the process of resolving. Despite these patent obstacles, we are optimistic with regard to providing a meaningful analysis of both problems and proceeding with the experimental determination which we have outlined.

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Fig. 1. Kinematical Model.

Fig. 2. Contacting Beams.

A FORMULATION OF THE MECHANICAL BEHAVIOR OF ATJ GRAPHITE AT HIGH TEMPERATURE

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ABSTRACT

The application of structural graphite to ballistic re-entry vehicle nose tips is current. Thermal stresses are a primary consideration for these designs. Generally design analyses are restricted to assumed linear elastic behavior, even though it is obvious from test data that the behavior is neither linear nor elastic at temperatures in excess of 4000°F. A formulation of the mechanical behavior appropriate for the above application is discussed.

Generally design data is obtained from uniaxial tensile tests at a standard cross-head speed. Representative stress-strain curves appear consistent with a plasticity (rate independent) formulation. Alternatively available high temperature creep data suggests the need for a rate dependent viscoelastic formulation.

A formulation which contradicts neither data is desirable. The plasticity formulation, which is rate independent, cannot agree with the creep data. In this talk a viscoelastic constitutive law is formulated on the basis of the creep data. Using this formulation, the response of a tensile specimen subjected to constant strain rate is predicted. The predicted response is consistent with both types of available data.

Although the viscoelastic law was formulated on the basis of creep data, the material parameters may be evaluated from other types of test data. For this reason, the response of other test specimens is predicted and methods for evaluating the material parameters are identified.

NOMENCLATURE

i, j	subscripts which may take the values 1, 2, or 3.
ϵ_{ij}	components of the strain tensor
σ_{ij}	components of the stress tensor
ϵ_{kk}	trace of the strain tensor, $= \epsilon_{11} + \epsilon_{22} + \epsilon_{33}$
σ_{kk}	trace of the stress tensor, $= \sigma_{11} + \sigma_{22} + \sigma_{33}$
$\bar{\epsilon}_{ij}$	deviatoric components of the strain tensor, $= \epsilon_{ij} - \epsilon_{kk}/3 \delta_{ij}$
$\bar{\sigma}_{ij}$	deviatoric components of the stress tensor $= \sigma_{ij} - \sigma_{kk}/3 \delta_{ij}$
ϵ^T	thermal expansion
δ_{ij}	Kronecker; $= 1$ if $i = j$; $= 0$ if $i \neq j$
T	temperature
t	time
J	shear creep function of linear viscoelasticity
K	bulk modulus
A, B, β	material parameters
J_2	second invariant of the stress deviator
α	scaler function of J_2
a	constant strain rate during a tensile test
ϵ_0	constant value of strain during a relaxation tensile test
σ_0	constant value of stress during creep test in uniaxial strain
τ_0	constant value of shear stress during a shear creep test
U	dummy variable of integration
$()$	time rate, $= \partial/\partial t ()$ for infinitesimal deformations

INTRODUCTION

In present structural design analyses the mechanical behavior of ATJ Graphite is assumed linear elastic. The major consideration in these evaluations is the possibility of rupture due to thermal stress, and therefore the assumption of linear elastic behavior is presumed conservative. This is a useful approach only when the design evaluation yields positive margins of safety. Alternatively, if positive margins are not evident on the basis of this approach, it still may be possible to show the design good on the basis of an improved formulation. Specifically the constitutive laws for the material at high temperature are most suspect. That the behavior of ATJ is not linear elastic (or even elastic) at temperatures in excess of 4000°F is obvious from most test data. What is a reasonable formulation for the behavior is not so obvious. Two potential formulations are plasticity and viscoelasticity. In this report a viscoelastic formulation is offered as appropriate.

There is another design consideration for homogeneous graphite nose tips which requires an improved constitutive law more than thermal stress evaluations, and that is the stability of nose configurations. Homogeneous graphites are not good insulators and a substantial amount of the nose tip is driven to temperatures in excess of 4000°F during ballistic re-entry. The possibility of material flow at these temperatures combined with the thermal expansion and severe aerodynamic pressure gradients may lead to excessive nose tip deformations.

These design considerations are the impetus for the study reported here.

AVAILABLE TEST DATA AND POTENTIAL FORMULATIONS

The conventional test for mechanical properties is a tensile (uniaxial stress) test. It is usual that this test be run at a standard cross-head speed (0.02 in/min.) which, for a given specimen configuration, may be equivalent to a constant strain rate test. While this test is completely sufficient for linear elastic behavior, it is insufficient to define a behavior that is not linear elastic.

Most design data (Young's modulus, Poisson's ratio, ultimate tensile strength) are obtained from tensile tests. A representative stress-strain curve for ATJ is presented by Figure 1¹. Obviously this data cannot be approximated by a linear elastic law. With only similar data available, an appropriate constitutive law is apparently within the framework of plasticity (rate independent). There is no obvious reason to believe that a plastic formulation cannot explain the available tensile data.

That a viscoelastic formulation (rate dependent) may be more appropriate becomes apparent only when other data are reviewed. In this case the other data are creep data that was obtained by AFML²⁻⁵ and brought to the writers attention by Dr. J. Gebhart - SSL. Typical creep data⁴ are represented by Figure 2. Of course, these data are strain as a function of time when the stress is held constant. The theory of plasticity cannot explain this behavior, since the data are time dependent. It appears natural to formulate the time dependent behavior by a viscoelastic constitutive law.

A single constitutive law that can explain both constant strain rate data and creep data is desirable. In the remainder of this report, a viscoelastic law is formulated on the basis of the creep data and shown to agree with constant strain rate data.

LINEAR VISCOELASTIC FORMULATION

It is common to assume that the bulk and shear behavior of a material are not coupled and furthermore that the bulk behavior is elastic. Making these assumptions, the constitutive relations for a linear viscoelastic material are

$$\epsilon_{kk} = \frac{\sigma_{kk}}{3K_T} + 3\epsilon^T \quad (1)$$

$$\bar{\epsilon}_{ij}(t) = \int_{-\Delta}^t J_T(t-\tau) \frac{\partial \bar{\sigma}_{ij}(\tau)}{\partial \tau} d\tau$$

where the subscript "T" on the bulk modulus "K" and the shear creep function "J" indicate the (constant) temperature at which they are measured.

For a creep test in uniaxial tension

$$\begin{bmatrix} \sigma_{ij} \end{bmatrix} = \begin{bmatrix} \sigma_{11} & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{bmatrix}; \quad \begin{bmatrix} \bar{\sigma}_{ij} \end{bmatrix} = \begin{bmatrix} \frac{2}{3} \sigma_{11} & 0 & 0 \\ 0 & -\frac{\sigma_{11}}{3} & 0 \\ 0 & 0 & -\frac{\sigma_{11}}{3} \end{bmatrix}$$

$$\sigma_{kk} = \sigma_{11} \quad (2)$$

and when Eq. (1) is solved for the strain in the direction of loading we obtain

$$\begin{aligned} \epsilon_{11}(t) &= \bar{\epsilon}_{11} + \frac{\epsilon_{kk}}{3} \\ &= \left(\frac{2}{3} J_T(t) + \frac{1}{9K_T} \right) \sigma_{11} \end{aligned} \quad (3)$$

Then the shear creep function is

$$J_T(t) = \frac{3}{2} \left[\frac{\epsilon_{11}(t)}{\sigma_{11}} - \frac{1}{9K_T} \right] \quad (4)$$

Therefore one can evaluate the shear creep function from tensile creep data (such as presented in Figure 2) if the bulk modulus is known. Also a serious limitation of linearity is evident in Eq. (4); since J_T is independent of the stress the function $\epsilon_{11}(t)/\sigma_{11}$ measured during a tensile creep test must be independent of the magnitude of σ_{11} used in the test. This restriction is violated by the creep data reported by AFML. The advantages of a linear viscoelastic formulation (one of which is the direct measure of the shear creep function – not requiring functional “fits” of the data) are lost when one must account for non-linearities.

NON-LINEAR FORMULATION

Four one-dimensional equations⁴ were fitted to the tensile creep data. The primary interest of the researchers doing this work was a determination of the activation energy, and their attention was focused on steady-state creep rates. They conclude that the creep rate varies as the fourth power of the stress:

$$\dot{\epsilon}_{11} \propto \sigma_{11}^4 \quad (\text{steady-state}) \quad (5)$$

This dependence is what invalidates the linear formulation.

They also indicate that the instantaneous elastic response (indicated by the values of the strain at zero time on Figure 2) appears proportional to the stress. These data are obtained by extrapolating back to zero time, since the loading is not instantaneous.

A generalization of the linear viscoelastic law represented by Eq. (1) to include nonlinearity is not unique. There are an infinitude of nonlinear constitutive laws which reduce to Eq. (1) for infinitesimal strains and rates. Also the generalization of the linear law over the complete time period represented by Figure 2 (90 minutes) would be very difficult and require a very comprehensive test program. Fortunately we don't require such a complete generalization for ballistic applications. Our attention can be focused on the behavior within the first couple of minutes of loading.

The tensile creep data within the first couple of minutes can be approximated by straight lines. If we assume the short time creep rate varies as some power of the stress (similar to the variation found for the steady-state creep rates but not necessarily the same power) the following one-dimensional law represents a short time approximation

$$\epsilon_{11}(t) = A_1 \sigma_{11} + B_1 t \sigma_{11}^\beta \quad (6)$$

where A_1 , B_1 , and β are parameters which may be temperature dependent but are independent of the stress or strain magnitudes.

A generalization of the above one-dimensional equation to account for unsteady temperature and stress histories is as follows:

$$\dot{\epsilon}_{11}(t) = \frac{\dot{\sigma}_{11}}{A_1} + B_1 \sigma_{11}^\beta \quad (7)$$

This represents probably the simplest generalization of Eq. (6). Higher time rates of stress could be added to Eq. (7), and it would still reduce to Eq. (6) for the case when it is appropriate – constant stress. Presently there is no data to warrant such complications.

THREE-DIMENSIONAL GENERALIZATION

The next step is to define a nonlinear generalization of Eq. (1), which is three-dimensional. Also the three-dimensional generalization should reduce to Eq. (7) for the case of uniaxial stress.

One such generalization follows:

$$\begin{aligned}\epsilon_{kk} &= \frac{\sigma_{kk}}{3K} + 3\epsilon^T \\ \dot{\bar{\epsilon}}_{ij} &= \underbrace{\dot{A} \bar{\sigma}_{ij}} + B \alpha(J_2) \bar{\sigma}_{ij}\end{aligned}\quad (8)$$

where A, B, and K are functions of temperature, J_2 is the second invariant of the stress deviator

$$J_2 = \frac{1}{2} \bar{\sigma}_{ij} \bar{\sigma}_{ij} \quad (9)$$

and $\alpha(J_2)$ is a function of J_2 (also the temperature), which is taken as follows for agreement with Eq. (7):

$$\alpha(J_2) = \frac{\beta - 1}{(3J_2)^2} \quad (10)$$

This generalization also is probably the simplest one consistent with the previous discussion.

For future use, the constitutive equations are written in terms of the total components of the stress and strain tensors:

$$\begin{aligned}\dot{\epsilon}_{11} &= \overbrace{\left[\left(\frac{2}{3} A + \frac{1}{9K} \right) \sigma_{11} - \left(A - \frac{1}{3K} \right) \frac{\sigma_{22} + \sigma_{33}}{3} \right]}^{\dot{A} \bar{\sigma}_{11}} \\ &\quad + B \alpha(J_2) \left[\frac{2}{3} \sigma_{11} - \frac{\sigma_{22} + \sigma_{33}}{3} \right] + \dot{\epsilon}^T \\ \dot{\epsilon}_{12} &= \underbrace{\dot{A} \bar{\sigma}_{12}} + B \alpha(J_2) \sigma_{12} \\ &\vdots\end{aligned}\quad (11)$$

and:

$$J_2 = \frac{1}{6} \left[(\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2 \right] + \sigma_{12}^2 + \sigma_{23}^2 + \sigma_{31}^2 \quad (12)$$

The equation for the other normal or shear components of the strain rate tensor are obtained from the two given explicitly by a permutation of the indices. The above equations are the generalized constitutive equations required for nose tip deformation or thermal stress analysis.

OTHER TEST SPECIMENS

Although the constitutive law, Eqs. (8) or (11), has been formulated qualitatively on the basis of tensile creep data, such testing may not be the best way to evaluate the required parameters. The response of other test specimens are now considered.

Uniaxial Stress

For uniaxial stress applied in the "1" direction, the strain rate in the same direction is obtained from Eq. (11):

$$\dot{\epsilon}_{11} = \left[\left(\frac{2}{3} A + \frac{1}{9K} \right) \sigma_{11} \right] + B \alpha (J_2)^{\frac{2}{3}} \sigma_{11} \quad (13)$$

From Eq. (12)

$$J_2 = \frac{\sigma_{11}^2}{3} \quad (14)$$

and with Eq. (10), Eq. (13) becomes

$$\dot{\epsilon}_{11} = \left[\left(\frac{2}{3} A + \frac{1}{9K} \right) \sigma_{11} \right] + \frac{2}{3} B \sigma_{11}^{\beta} \quad (15)$$

This equation is identical to Eq. (7) — as it should be — if A_1 is associated with $2/3A + 1/9K$ and B_1 with $2/3B$.

Constant Strain Rate

By far, the most data that is accumulated for design is constant strain rate (constant cross-head motion) tensile data. If we take "a" as the strain rate and recognize that "A", "K", "B", and " β " are fixed during a test conducted at constant temperature, the above equation becomes a differential equation for the resulting stress history:

$$\dot{\sigma}_{11} + \frac{B}{A + \frac{1}{6K}} \sigma_{11}^{\beta} - \frac{3/2 a}{A + \frac{1}{6K}} = 0 \quad (16)$$

This can be solved for strain versus stress:

$$\epsilon_{11} = \left(\frac{2}{3} A + \frac{1}{9K} \right) \left(\frac{3}{2} \frac{a}{B} \right)^{1/\beta} \int_0^{\left(\frac{2}{3} \frac{B}{a} \right)^{1/\beta} \sigma_{11}} \frac{dy}{1-y^\beta} \quad (17)$$

Predicted stress-strain relations are presented on Figure 3 for values of β of 1, 2, 3, and 4. It is noted that the predicted stress is bounded by the magnitude $(3a/2B)^{1/\beta}$

$$\sigma_{11 \text{ max}} = \left(\frac{3}{2} \frac{a}{B} \right)^{1/\beta} \quad (18)$$

Of course these predictions are limited to the first couple of minutes.

The curves predicted on the basis of viscoelastic behavior bear a close resemblance to measured data. The "apparent yield" is quite obvious for high values of β .

Eq. (7) is now analyzed to get a method for evaluating the parameters in the constitutive equation. First the initial tangent modulus of the stress-strain curve is

$$\frac{d \sigma_{11}}{d \epsilon_{11}} (0) = \frac{1}{\left(\frac{2}{3} A + \frac{1}{9K} \right)} \quad (19)$$

which is independent of the strain rate "a" and the values of "B" and " β ". Therefore the value of $(2A/3 + 1/9K)$ can be evaluated from simply the initial tangent modulus of the stress-strain data.

The tangent modulus for any value of stress also can be computed from Eq. (17):

$$\frac{d \sigma_{11}}{d \epsilon_{11}} = \frac{1}{\left(\frac{2}{3} A + \frac{1}{9K} \right)} \left[1 - \frac{2B}{3a} \sigma_{11}^\beta \right] \quad (20)$$

Upon rearranging and taking the logarithm

$$\log \left[1 - \left(\frac{2}{3} A + \frac{1}{9K} \right) \frac{d \sigma_{11}}{d \epsilon_{11}} \right] = \beta \log \sigma_{11} + \log \left(\frac{2B}{3a} \right) \quad (21)$$

Therefore if $[1 - (2/3A + 1/9K) d\sigma_{11}/d\epsilon_{11}]$ is plotted versus σ_{11} on a log-log scale the data should fall on a straight line. The slope of the line gives a value for β and $2B/3a$ is evaluated from its intercept. To evaluate "B" and "A" the values of "a" (the strain rate) and "K" (the bulk modulus) must be known. "a" must be measured during the constant strain rate test. "K" can be evaluated from a uniaxial strain test, which will be discussed, or a different combination of "K" and "A" can be determined from the same tensile test by measuring the transverse strain. From Eq. (11)

$$\dot{\epsilon}_{22} = - \left(A - \frac{1}{3K} \right) \frac{\dot{\sigma}_{11}}{3} - B \frac{\sigma_{11}^\beta}{3} \quad (22)$$

From this equation it follows that the initial tangent modulus of σ_{11} versus ϵ_{22} is

$$\frac{d\sigma_{11}}{d\epsilon_{22}} (o) = - \frac{3}{A - \frac{1}{3K}} \quad (23)$$

Eqs. (19) and (23) can be solved simultaneously for values of "A" and "K":

$$\begin{aligned} A &= \frac{1}{\frac{d\sigma_{11}}{d\epsilon_{11}} (o)} - \frac{1}{\frac{d\sigma_{11}}{d\epsilon_{22}} (o)} \\ \frac{1}{3K} &= \frac{1}{\frac{d\sigma_{11}}{d\epsilon_{11}} (o)} + \frac{2}{\frac{d\sigma_{11}}{d\epsilon_{22}} (o)} \end{aligned} \quad (24)$$

This approach for evaluating the material parameters (short of separating "A" and "K") was applied to the constant strain rate tensile data presented on Figure 1. The log-log plot representing Eq. (21) is shown by Figure 4. A straight line represents the data well up to $\sigma_{11} \approx 4 \times 10^3$ psi ($\log \sigma_{11} = 3.6$). Looking back at Figure 1, this represents $\epsilon_{11} \approx 0.02$ in/in. The cross-head speed during the test was the standard 0.02 in/min. A specific strain-time trace is not available for this data, but it is estimated that this level of strain would be reached at the end of two minutes of loading. The constitutive law presented in this report is also restricted to the same short time and therefore the data is not inconsistent with the formulation.

Using the parameters just evaluated in Eq. (17), a predicted stress-strain curve was developed. The predictions are plotted as "X's" on the same experimental curve and presented on Figure 5. The agreement between predicted and measured data within the first two minutes is good. Measured data at greater rates would offer a more complete evaluation of the short time constitutive law.

Relaxation

In a relaxation test one imposes a strain on the tensile specimen and measures the decay of stress with time. The strain history is

$$\epsilon_{11} = \begin{cases} 0 & t < 0 \\ \epsilon_o & t > 0 \end{cases} \quad (25)$$

For

$$t > 0$$

$$\dot{\epsilon} = 0$$

and Eq. (15) becomes

$$\dot{\sigma}_{11} + \frac{B}{A + \frac{1}{6K}} \sigma_{11}^{\beta} = 0 \quad (26)$$

A solution to this equation follows:

$$\frac{\sigma_{11}(t)}{\sigma_{11}(0)} = \left\{ 1 + \frac{B}{A + \frac{1}{6K}} (\beta - 1) \sigma_{11}(0)^{\beta-1} t \right\}^{\frac{1}{\beta-1}} \quad (27)$$

for $\beta > 1$ and $t \geq 0$

and where $\sigma_{11}(0)$ is the initial value of σ_{11} . From the solution no particular advantage of relaxation testing is obvious.

Uniaxial Strain

In addition to uniaxial stress the state of uniaxial strain also offers some analysis advantages – particularly for the evaluation of the bulk modulus. This state of strain is not so easily attained but is usually approximated by testing a wide plate subject to loading (uniformly) in the direction of its small dimension (thru-the-thickness).

If we take the “1” direction to be the small dimension and the loading direction, the stress and strain tensors are

$$\epsilon_{ij} = \begin{bmatrix} \epsilon_{11} & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{bmatrix} ; \quad \bar{\epsilon}_{ij} = \begin{bmatrix} \frac{2}{3}\epsilon_{11} & 0 & 0 \\ 0 & -\frac{\epsilon_{11}}{3} & 0 \\ 0 & 0 & -\frac{\epsilon_{11}}{3} \end{bmatrix} \quad (28)$$

$$\sigma_{ij} = \begin{bmatrix} \sigma_{11} & 0 & 0 \\ 0 & \sigma_{22} & 0 \\ 0 & 0 & \sigma_{22} \end{bmatrix} ; \quad \bar{\sigma}_{ij} = \begin{bmatrix} \frac{2}{3}(\sigma_{11} - \sigma_{22}) & 0 & 0 \\ 0 & -\frac{(\sigma_{11} - \sigma_{22})}{3} & 0 \\ 0 & 0 & -\frac{(\sigma_{11} - \sigma_{22})}{3} \end{bmatrix}$$

and other required quantities are

$$\begin{aligned} \epsilon_{kk} &= \epsilon_{11} \\ \sigma_{kk} &= (\sigma_{11} - 2\sigma_{22}) \\ 3J_2 &= (\sigma_1 - \sigma_2)^2 \end{aligned} \quad (29)$$

For the constitutive relations it is convenient to use Eq. (8), which becomes

$$\begin{aligned}\epsilon_{11} &= \frac{\sigma_{11} + 2\sigma_{22}}{3K} \quad (\text{Bulk}) \\ \dot{\epsilon}_{11} &= \underbrace{A(\sigma_{11} - \sigma_{22})}_{\text{Bulk}} + B(|\sigma_{11} - \sigma_{22}|)^{\beta-1}(\sigma_{11} - \sigma_{22}) \quad (\text{Deviator})\end{aligned}\quad (30)$$

Creep

To consider a creep test we take

$$\sigma_{11} = \sigma_o \text{ constant for } t > 0 \quad (31)$$

and use the first of Eq. (30) to eliminate σ_{22} from the second:

$$\dot{\epsilon}_{11} = \frac{3}{2} A (\sigma_o - K\epsilon_{11}) + \left(\frac{3}{2}\right)^{\beta} B (|\sigma_o - K\epsilon_{11}|)^{\beta-1} (\sigma_o - K\epsilon_{11}) \quad (32)$$

An equilibrium position will exist at large time:

$$\begin{aligned}\epsilon_{11} &\longrightarrow \frac{\sigma_o}{K} \\ t &\longrightarrow \infty\end{aligned}\quad (33)$$

Although the constitutive law used to deduce this result is based on a short time approximation, the result presented by Eq. (33) is valid. The data for ATJ at high temperatures show that a steady state creep exists in tension and therefore the equilibrium state in uniaxial strain is a hydrostatic stress state. A practical question of how much time is required to attain equilibrium is best answered by experiment.

To predict the short time response, Eq. (32) is rearranged:

$$\left(1 + \frac{3}{2} AK\right) \dot{\epsilon}_{11} = \left(\frac{3}{2}\right)^{\beta} B (|\sigma_o - K\epsilon_{11}|)^{\beta-1} (\sigma_o - K\epsilon_{11}) \quad (34)$$

This equation can be solved by separating variables:

$$t = \left(\frac{2}{3}\right)^{\beta} \frac{(1 + \frac{3}{2} AK)}{B} \int_{\epsilon_{11}(0^+)}^{\epsilon_{11}(t)} \frac{du}{(|\sigma_o - Ku|)^{\beta-1} (\sigma_o - Ku)} \quad (35)$$

This solution is not particularly convenient but it can be tabulated if required for data analysis.

If the short time history of ϵ_{11} is measured then ϵ_{11} can be computed at any given time. If we evaluate Eq. (34) at a specific time, say t_1 , we get the following:

$$\frac{3}{2}AK = \frac{\left(\frac{3}{2}\right)^\beta B (|\sigma_o - K\epsilon_{11}(t_1)|)^{\beta-1} [\sigma_o - K\epsilon_{11}(t_1)]}{\dot{\epsilon}_{11}(t_1)} - 1 \quad (36)$$

If values of "B" and " β " are available (from tensile data) the above equation can be solved simultaneously with Eq. (19) for values of "A" and "K".

Pure Shear (Torsion)

The most direct measure of "A", "B", and " β " is in the state of pure shear. For this state, the single nontrivial equation from Eq. (11) is

$$\dot{\epsilon}_{12} = A\sigma_{12} + (3)^{\frac{\beta-1}{2}} B\sigma_{12}^\beta \quad (37)$$

where Eqs. (10) and (12) have been used and σ_{12} is assumed positive.

Creep

If we let

$$\sigma_{12} = \tau_o \text{ for } t > 0 \quad (38)$$

Eq. (37) has the following solution:

$$\dot{\epsilon}_{12} = A\tau_o + (3)^{\frac{\beta-1}{2}} B\tau_o^\beta t \quad (39)$$

"A" can be evaluated from the instantaneous elastic response and "B" and " β " can be evaluated from the short time creep rates for different levels of τ_o .

Constant Strain Rate

To consider constant strain rate testing we let

$$\dot{\epsilon}_{12} = \gamma \text{ a constant} \quad (40)$$

Eq. (37) then becomes

$$\dot{\sigma}_{12} + (3)^{\frac{\beta-1}{2}} \left(\frac{B}{A}\right) \sigma_{12}^\beta - \frac{\gamma}{A} = 0 \quad (41)$$

This equation is similar to Eq. (16) for uniaxial stress. The solution for strain versus stress is also similar:

$$\epsilon_{12} = \frac{A(\gamma/B)^{1/\beta}}{(3)^{\frac{\beta-1}{2\beta}}} \int_0^{(3)^{\frac{\beta-1}{2\beta}} (B/\gamma)^{\frac{1}{\beta}} \sigma_{12}} \frac{dy}{1-y^\beta} \quad (42)$$

An analysis of this equation appropriate for data interpretation can be performed as it was for the case of uniaxial stress.

Solution Techniques

To study mechanical deformations and/or thermal stresses in nose tips, a numerical solution and computer program can be developed. Of course equations other than the constitutive law are required. For this discussion, these other equations are the equations of motion (equilibrium equations) and the compatibility relations.

Although it may not be the most accurate approach, it is expedient that an analogy can be established between the response of a viscoelastic nose tip and an elastic one. Since computer programs for the elastic case are available they can be used to evaluate the viscoelastic response.

Of the governing equations, only the constitutive equations are effected by the different material behavior. The equilibrium and compatibility equations are identical, whatever the material behavior. Therefore for the development of the analogy only the constitutive equations need be considered.

Consider the values of the stress and strain tensors at two instants of time, t and $t + \Delta t$, separated by a small interval Δt . Also, the difference in the value of a function f at these two instants is denoted by $\Delta f(t)$:

$$\Delta f(t) = f(t + \Delta t) - f(t) \quad (43)$$

If we evaluate the first of Eq. (8) at $t + \Delta t$ and t , and then subtract them, we obtain the following:

$$\Delta \epsilon_{kk}(t) = \frac{\Delta \sigma_{kk}(t)}{3K(t + \Delta t)} + \left[\frac{1}{K(t + \Delta t)} - \frac{1}{K(t)} \right] \frac{\sigma_{kk}(t)}{3} + 3\Delta \epsilon^T(t) \quad (44)$$

For the rates in the second of Eq. (8) we use forward differences.

$$\begin{aligned}\dot{\bar{\epsilon}}_{ij}(t) &\approx \frac{\Delta \bar{\epsilon}_{ij}(t)}{\Delta t} \\ \dot{\bar{\sigma}}_{ij}(t) &\approx \frac{A(t + \Delta t) \bar{\sigma}_{ij}(t + \Delta t) - A(t) \bar{\sigma}_{ij}(t)}{\Delta t} \\ &\approx \frac{A(t + \Delta t) \Delta \bar{\sigma}_{ij}(t)}{\Delta t} + \frac{\Delta A(t) \bar{\sigma}_{ij}(t)}{\Delta t}\end{aligned}\quad (45)$$

Using these difference approximations the shear constitutive equation becomes the following:

$$\begin{aligned}\Delta \bar{\epsilon}_{ij}(t) &= A(t + \Delta t) \Delta \bar{\sigma}_{ij}(t) \\ &+ \left[\Delta A(t) + B(t) \alpha(J_2, t) \Delta t \right] \bar{\sigma}_{ij}(t)\end{aligned}\quad (46)$$

Eqs. (44) and (46) are in the form appropriate for the analogy. They are interpreted as equations relating the increments of the stress and strain tensors when the values of the stress and strain tensors are known at time t . When the above equations are written in component form they become

$$\begin{aligned}\Delta \epsilon_{aa}(t) &= \frac{1}{E(t + \Delta t)} \left\{ \Delta \sigma_{aa}(t) - \nu(t + \Delta t) [\Delta \sigma_{bb}(t) + \Delta \sigma_{cc}(t)] \right\} + \epsilon_{aa}^T(t) \\ \vdots \\ \Delta \epsilon_{ab}(t) &= \frac{1}{2G(t + \Delta t)} \Delta \sigma_{ab}(t) + \epsilon_{ab}^T(t) \\ \vdots\end{aligned}\quad (47)$$

where the equivalent Young's modulus and Poisson's ratio are defined as

$$\begin{aligned}E(t + \Delta t) &= \frac{9 K(t + \Delta t)}{1 + 6 A(t + \Delta t) K(t + \Delta t)} \\ \nu(t + \Delta t) &= \frac{1}{3} \left[A(t + \Delta t) - \frac{1}{3 K(t + \Delta t)} \right] E(t + \Delta t)\end{aligned}\quad (48)$$

and the equivalent thermal expansion is

$$\begin{aligned}\epsilon_{aa}^T(t) &= \Delta \epsilon^T(t) + \left[\frac{1}{K(t + \Delta t)} - \frac{1}{K(t)} \right] \frac{\sigma_{aa}(t) + \sigma_{bb}(t) + \sigma_{cc}(t)}{9} \\ &+ [\Delta A(t) + B(t) \alpha(J_2, t) \Delta t] \frac{1}{3} [2 \sigma_{aa}(t) - \{\sigma_{bb}(t) + \sigma_{cc}(t)\}]\end{aligned}\quad (49)$$

$$\begin{aligned}\epsilon_{ab}^T(t) &= [\Delta A(t) + B(t) \alpha(J_2, t) \Delta t] \sigma_{ab}(t) \\ \vdots \\ \vdots\end{aligned}$$

Eq. (47) is identical to the stress-strain relations for a linear elastic material whose mechanical properties are isotropic and whose thermal expansion is orthotropic. Therefore Eqs. (48) and (49) establish the desired analogy to use existing elastic solutions to study the viscoelastic response. The duration of the problem to be studied is divided into short time intervals and the increments in stresses and strains are computed using the analogous elastic solution. The total stress or strain at any time is then determined by summing the incremental values up to that time.

CONCLUSIONS

A viscoelastic constitutive relation has been formulated for the behavior of ATJ graphite at high temperatures. The rate dependence is not apparent in design data because these tests are conducted at standard test rates. Tests must be designed specially to evaluate the rate dependence for short time. Application studies should be performed to determine the sensitivity of representative designs to the rate dependence and establish the need for further experimental evaluations.

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Fig. 1. Tensile Stress vs Strain, ATJ Graphite, With Grain, 5000°F.

Fig. 2. Creep of ATJ Graphite.

Fig. 3. Predicted Behavior for Constant Strain Rate.

Fig. 4. Material Parameter Evaluation.

Fig. 5. Comparison of Experimental and Predicted Behavior, ATJ Graphite, With Grain, 5000°F.

✓ VISCOELASTIC REPRESENTATION OF ANISOTROPIC MECHANICAL BEHAVIOR
OF GRAPHITE AT ELEVATED TEMPERATURES*

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ABSTRACT

As a result of fabrication processes and other factors, the mechanical behaviors of many graphites exhibit varying degrees of anisotropy. At elevated temperatures, creep and relaxation mechanisms are operative and give rise to mechanical behavior which is both time and temperature dependent. These are some of the features which must be taken into account when developing constitutive equations applicable to these materials.

A mathematical characterization of the time-dependent mechanical behavior of RVD graphite is discussed here for temperatures ranging from 3500 to 5000°F. A set of temperature-dependent constitutive equations is shown to describe the mechanical response of the material to a variety of loadings. Experimental results are used both to numerically evaluate parameters and to verify the resulting mathematical model.

First, RVD graphite, a molded graphite, is characterized as transversely isotropic with the plane of isotropy normal to the direction of molding. For stress levels up to one half of the nominal ultimate strength of the material, creep strains are observed to have linear dependences upon stresses.

The constitutive equations used represent a transversely isotropic linear viscoelastic model and are formulated in terms of hereditary integrals. These relations contain five independent material properties: three strain ratios (Poisson's ratios) and two creep functions. The strain ratios are temperature dependent, but independent of time, stress, and strain. Thus, the time-dependent character of the behavior is accounted for by two creep functions. Four of the five parameters were first evaluated at 4500°F by using data from with-grain (directions parallel to plane of isotropy) and across-grain (direction normal to the plane of isotropy) tensile tests and from with-grain creep tests. Temperature dependence is introduced into the model by considering data from with-grain tensile and creep tests conducted at 3500, 4000, and 5000°F. The experimental data on which this study is based did not provide information for the determination of the creep function which describes the shear behavior in planes normal to the plane of isotropy. The model as developed, however, allows predictions for a wide variety of problems which do not involve this type of shear behavior. Among these are plane strain, plane stress, and axisymmetric problems, when in each case the defining plane is the plane of isotropy.

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To provide a degree of verification, predictions based on these constitutive equations are shown to closely agree with results of across-grain tensile creep tests, with-grain and across-grain relaxation tests, and short time stress-strain behavior for three different strain rates. The good agreement found between the predictions and experimental results is significant, as these results are independent of those used in numerically evaluating the parameters in the constitutive equations.

In many structural applications of graphite, the designer must deal with complex loading conditions and complicated geometries for which he will not have directly applicable test data. We will describe the development of a unified set of constitutive equations capable of providing reasonable predictions of data from tests different from those used in the development of the equations themselves. This development involves considerably more than the fitting of an equation to data from a single type of laboratory test.

The experimental data used in this study were provided by the Southern Research Institute, and were results of uniaxial creep, relaxation, and constant strain rate tensile tests of RVD graphite. RVD is a molded nuclear grade graphite having characteristic transverse isotropic behavior. The molding direction is the across-grain direction. The with-grain directions are normal to the direction of molding. Both with-grain and across-grain specimens were tested at temperatures of 3500, 4000, 4500, and 5000°F. As illustrated by the uniaxial with-grain creep curves in Fig. 1, the mechanical deformation processes are time dependent over the prescribed temperature range. An examination of the creep strains developed in such tests at a specific time provides insight into the dependence of the time-dependent behavior on stress level. With the specified time taken as 20 minutes, the plot in Fig. 2 provides an indication that for a temperature of 4500°F the behavior depends linearly on stress for values up to 3700 psi, approximately 50% of the with-grain ultimate strength of the material. This same range of linearity was observed for other temperatures as well. Therefore, a temperature-dependent transversely isotropic linear visco-elastic model was employed within the prescribed stress limitation.

The constitutive equations are expressed in a form exhibiting an axis of rotational symmetry. The plane of isotropy is defined by the rectangular Cartesian coordinates (x_1, x_2) , and the direction normal to this plane will be represented by x_3 , the molding direction. The basic constitutive equations are expressed in terms of hereditary integrals:

$$\begin{aligned}\epsilon_{11}(t) &= \int_0^t J_{11}(t - \tau) \left[\frac{\partial \sigma_{11}}{\partial \tau} - \mu_{12} \frac{\partial \sigma_{22}}{\partial \tau} - \mu_{13} \frac{\partial \sigma_{33}}{\partial \tau} \right] d\tau, \\ \epsilon_{22}(t) &= \int_0^t J_{11}(t - \tau) \left[\frac{\partial \sigma_{22}}{\partial \tau} - \mu_{12} \frac{\partial \sigma_{11}}{\partial \tau} - \mu_{13} \frac{\partial \sigma_{33}}{\partial \tau} \right] d\tau, \\ \epsilon_{33}(t) &= \frac{\mu_{13}}{\mu_{31}} \int_0^t J_{11}(t - \tau) \left[\frac{\partial \sigma_{33}}{\partial \tau} - \mu_{31} \frac{\partial}{\partial \tau} (\sigma_{11} + \sigma_{22}) \right] d\tau, \\ \epsilon_{23}(t) &= \frac{1}{2} \int_0^t J_{44}(t - \tau) \frac{\partial \sigma_{23}}{\partial \tau} d\tau, \\ \epsilon_{13}(t) &= \frac{1}{2} \int_0^t J_{44}(t - \tau) \frac{\partial \sigma_{13}}{\partial \tau} d\tau, \\ \epsilon_{12}(t) &= (1 + \mu_{12}) \int_0^t J_{11}(t - \tau) \frac{\partial \sigma_{12}}{\partial \tau} d\tau.\end{aligned}$$

The μ_{ij} values are ratios of lateral strains induced in the x_j directions to the longitudinal strains produced by a uniaxial stress applied in the x_i directions. In the development, the strain ratios are assumed to be independent of time, stress, and strain. $J_{11}(t)$ and $J_{44}(t)$ are independent creep functions. By using Laplace transforms, the inverse constitutive equations can be developed:

$$\begin{aligned}\sigma_{11}(t) &= \beta \int_0^t G_{11}(t - \tau) \left[\frac{\partial \epsilon_{11}}{\partial \tau} + \alpha_1 \frac{\partial \epsilon_{22}}{\partial \tau} + \alpha_2 \frac{\partial \epsilon_{33}}{\partial \tau} \right] d\tau, \\ \sigma_{22}(t) &= \beta \int_0^t G_{11}(t - \tau) \left[\frac{\partial \epsilon_{22}}{\partial \tau} + \alpha_1 \frac{\partial \epsilon_{11}}{\partial \tau} + \alpha_2 \frac{\partial \epsilon_{33}}{\partial \tau} \right] d\tau, \\ \sigma_{33}(t) &= \beta \int_0^t G_{11}(t - \tau) \left[\alpha_3 \frac{\partial \epsilon_{33}}{\partial \tau} + \alpha_2 \frac{\partial}{\partial \tau} (\epsilon_{11} + \epsilon_{22}) \right] d\tau, \\ \sigma_{23}(t) &= 2 \int_0^t G_{44}(t - \tau) \frac{\partial \epsilon_{23}}{\partial \tau} d\tau, \\ \sigma_{13}(t) &= 2 \int_0^t G_{44}(t - \tau) \frac{\partial \epsilon_{13}}{\partial \tau} d\tau, \\ \sigma_{12}(t) &= \beta(1 - \alpha_1) \int_0^t G_{11}(t - \tau) \frac{\partial \epsilon_{12}}{\partial \tau} d\tau.\end{aligned}$$

As shown below, $G_{11}(t)$ and $G_{44}(t)$ are independent relaxation functions determined from the creep functions, while α_1 , α_2 , α_3 , and β are functions of the strain ratios:

$$\int_0^t J_{11}(t - \tau) G_{11}(\tau) d\tau = t H(t) ,$$

$$\int_0^t J_{44}(t - \tau) G_{44}(\tau) d\tau = t H(t) ,$$

$$\alpha_1 = \frac{\mu_{12} + \mu_{13} \mu_{31}}{1 - \mu_{13} \mu_{31}} ,$$

$$\alpha_2 = \frac{\mu_{31} (1 + \mu_{12})}{1 - \mu_{13} \mu_{31}} ,$$

$$\alpha_3 = \frac{\mu_{31} (1 - \mu_{12}^2)}{\mu_{13} (1 - \mu_{13} \mu_{31})} ,$$

$$\beta = \frac{1}{(1 - \mu_{12} \alpha_1 - \mu_{13} \alpha_2)} .$$

The five parameters to be evaluated for complete determination of the constitutive equations at a given temperature are:

$$\mu_{12}, \mu_{13}, \mu_{31},$$

$$J_{11}(t), J_{44}(t).$$

Each of these parameters was assumed to be temperature dependent. The first four parameters were evaluated from available experimental data. However, $J_{44}(t)$, which describes the shear behavior in planes normal to the plane of isotropy, could not be evaluated from the available data. This limitation does not prevent predictions of plane strain, plane stress, and axisymmetric problems which do not involve this type of shear behavior.

As the first step in the parameter evaluation (Fig. 3), average measured values of μ_{12} and μ_{31} were fitted in the sense of least squares by linear functions of the temperature. A cubic relationship was developed for μ_{13} .

Except for very short times, data from with-grain uniaxial creep tests were used to evaluate $J_{11}(t)$. A Maxwell model was used to describe the material behavior for the short times which correspond to typical loading intervals. The resulting creep function for the initial loading period is

$$J_{11}(t) = \frac{\mu_{13}}{\mu_{31}} \frac{1}{Ak} [1 + 0.02kt],$$

where A and k are material constants. The creep tests used specimens which were loaded at a constant strain rate (0.02 min^{-1}) to prescribed stress levels which were then held constant over a period of time. The entire creep function applicable for isothermal conditions and for all times up to the end of the period of data collection was taken to be of the following form:

$$J_{11}(t) = \frac{1}{E} + \eta t + \sum_{i=1}^3 A_i (1 - e^{-t/\tau_i}).$$

This may be interpreted in terms of (1) an elastic response, (2) a viscous component, and (3) a retarded elastic portion. The parameters were numerically evaluated at 4500°F first. The parameter E was measured by the initial slope of the with-grain stress-strain curve for a constant strain rate test (0.02 min^{-1}); η is the slope of the creep strain per unit stress versus time curve at the end of test. The remaining three terms were fitted in the sense of least squares so that $J_{11}(t)$ adequately represents the experimental data, as shown in Fig. 4, for example.

Temperature functions were next introduced so that the values given by the model for initial modulus, for duration of primary creep, and for creep strain and creep strain rate at the end of the with-grain tests agree with experimental results (e.g., Figs. 5 and 6). The resulting creep function is:

$$J_{11}(t, T) = \frac{\phi_0}{E} + A_1 \phi_1 (1 - e^{-\phi_3 t/\tau_1}) + \phi_2 \left[\eta \phi_4 t + A_2 (1 - e^{-\phi_3 t/\tau_2}) + A_3 (1 - e^{-\phi_3 t/\tau_3}) \right],$$

where the ϕ 's are functions of temperature and where E , η , A_1 , and τ_1 have the values that correspond to 4500°F. Specifically, the ϕ functions were determined to be

$$\begin{aligned}\phi_0(T) &= 1.64 T \times 10^{-4} + 0.186 , \\ \phi_1(T) &= 37.67 e^{-19.6 \times 10^3/RT} , \\ \phi_2(T) &= 2.32 e^{-7.58 \times 10^3/RT} + 3.16 \times 10^{10} e^{-135.1 \times 10^3/RT} , \\ \phi_3(T) &= 63.47 e^{-19.77 \times 10^3/RT} - 0.631 , \\ \phi_4(T) &= 12.96 \times 10^6 e^{-88.45 \times 10^3/RT} ,\end{aligned}$$

where T is the absolute temperature in degrees Rankine and R is the universal gas constant.

The temperature dependent model was subsequently used to predict across-grain creep behavior (e.g., Figs. 7 and 8). Good agreement was found between the predicted response and the average experimental data. Relaxation functions, which were determined from the creep functions through the use of Laplace transforms, were used to predict results of relaxation tests (e.g., Fig. 9). Excellent agreement between predicted and average experimental curves is seen for both with-grain and across-grain relaxation tests. Note that the ratio of these two relaxation curves vividly illustrates the degree of anisotropy present.

Finally, the model was used to predict the results of short-term stress-strain tests (e.g., Fig. 10) for the various temperatures. In order to investigate strain rate effects on short-time behavior, with-grain stress-strain tests were conducted at three distinct strain rates at 4500°F. Figure 11 shows that the strain rate effect is significant and that these effects are predicted very closely by the developed constitutive equations.

In most cases the predicted stress-strain curves deviate from test results for large stress values. It should be kept in mind, however, that the model was observed originally to be applicable only for stress levels up to 50% of the ultimate tensile strength of the material or approximately halfway up the experimental stress-strain curves.

In summary, this model has been shown to adequately predict uniaxial creep, relaxation, and stress-strain behaviors of both with-grain and across-grain specimens. The last two figures serve to indicate that the

range of applicability of the formulation is up to $1/2$ the ultimate tensile strength of the material. Refinements are planned in the form of developing a nonlinear viscoelastic model which will permit predictions of time-dependent behavior over the entire stress range. It is also planned to couple these formulations with elastic-plastic representations of time-independent behavior.

Fig. 1. RVD With-Grain Tensile Creep at 4500°F.

Fig. 2. With-Grain Creep Strain/Stress vs Stress at 4500°F.

Fig. 3. Strain Ratios as Functions of Temperature.

Fig. 4. RVD With-Grain Tensile Creep at 4500°F. Average experimental curve and calculated curve.

Fig. 5. RVD With-Grain Tensile Creep at 4000°F. Average experimental curve and calculated curve.

Fig. 6. RVD With-Grain Tensile Creep at 5000°F. Average experimental curve and calculated curve.

Fig. 7. RVD Across-Grain Tensile Creep at 4000°F. Average experimental curve and predicted curve.

Fig. 8. RVD Across-Grain Tensile Creep at 5000°F. Average experimental curve and predicted curve.

Fig. 9. RVD Stress Relaxation at 4500°F. Average experimental curves and predicted curves.

Fig. 10. Tensile Stress-Strain Curves for RVD Graphite. Average experimental curves and predicted curves.

Fig. 11. Effect of Strain Rate on RVD With-Grain Tensile Stress-Strain Curves at 4500°F.

✓ A Viscoelastic Representation of the Behavior of Graphite
Under the Influence of Neutron Irradiation*

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Abstract

In order to establish maximum design lifetimes for graphite core structures within reactor vessels, it is necessary to have a representative mathematical statement of material behavior. In particular, the constitutive equations must accommodate the effects of neutron exposure and provide a sound basis for methods of stress analysis. Toward this end, a viscoelastic representation of the mechanical behavior of graphite under neutron exposure is developed and is compared with experimental data for neutron-induced creep behavior. The mathematical model also includes provisions for the effects of neutron-induced swelling as well as thermal expansion.

When exposed to neutron irradiation, artificial graphites generally experience internal heat generation, induced creep, and dimensional changes which are functions of the accumulated neutron exposure and the irradiation temperature. It is shown that the linear viscoelastic model for thermorheologically simple materials is adaptable to the representation of experimental observations. In this model, the accumulated neutron exposure, which is in general nonuniform throughout the body, is identified as the "reduced time" variable. Temperature is included in the model as an additional parameter with no direct relation to the "reduced time," in contrast to

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the classical thermorheologically simple model. In addition, the constitutive equations are expressed in a form which characterizes a transversely isotropic material, since many nuclear-grade graphites exhibit this type of material symmetry as a result of fabrication processes.

The application of these constitutive equations to the solution of generalized plane-strain problems for isotropic materials is also discussed. It is shown that these viscoelastic solutions can be determined from associated elastic solutions.

INTRODUCTION

Graphite structural members in the core of a nuclear reactor experience an internal heat generation, exhibit creep and tend to change dimensions as functions of neutron exposure. At temperature levels of 500 to 1000°C currently being considered for the design of a molten salt breeder reactor (MSBR), the graphite initially contracts, reaches a minimum volume and then expands, as discussed by Kasten *et al.* [1].* The original volume is regained and the graphite continues to expand at an increasing rate for fast neutron dose values above about 3×10^{22} nvt ($E > 50$ kev)** until the structural integrity is destroyed. A reasonable design criteria would be to limit the operating life of the graphite structural elements by the fast neutron exposure (fluence), or time, required for the graphite to return to its original volume, provided that the stresses induced by differential dimensional changes within the structure remain low.

*The numbers in brackets refer to similarly numbered references at the end of this paper.

**Neutron dose is a measure of accumulated exposure to neutrons whose energy levels are greater than a certain threshold level, in this case 50×10^3 electron-volts ($E > 50$ kev). The unit "nvt" is an acronym for the product of "n," the neutron density (neutrons/cm³), times "v," the average neutron velocity (cm/sec), times "t" the exposure time (sec). The unit "nvt" thus has dimensions of neutrons/cm².

A viscoelastic representation of the mechanical behavior of irradiated graphite is developed in order to permit the evaluation of such design criteria and to provide a method of stress analysis. This representation is based upon the experimental results reported by Kennedy [2] which show that graphite under irradiation can be represented by a linear viscoelastic model if the accumulated neutron exposure (dose) is identified as a "reduced time" variable.

The constitutive equations for describing this material behavior are developed analogous to those for a thermorheologically simple material in the presence of nonuniform temperature distributions. The equations are initially expressed in a form to characterize a transversely isotropic material, since many nuclear grade graphites exhibit this type of material symmetry as a result of fabrication processes. The application of the constitutive equation to the solution of generalized plane-strain problems is also discussed.

CONSTITUTIVE EQUATIONS

Experiments [2] as shown in Figs. 1 and 2 indicate that the uniaxial creep curve for irradiated graphite approximates the linear superposition principle in such a way that the creep strain shown in Fig. 3 at neutron dose D_2 can be represented by a linear combination of the creep due to the stress σ_1 at zero dose and that due to an additional stress $-\sigma_2$ at D_1 as given by the following relation

$$\epsilon(D_2) = \sigma_1 J(D_2) - \sigma_2 J(D_2 - D_1) \quad (1)$$

where $J(D)$ is a dose-dependent creep function. Hence, for a continuously changing stress σ , the strain ϵ for an arbitrary accumulated dose D can be represented by the integral relation

$$\epsilon(D) = \int_0^D J(D - D') \frac{d\sigma}{dD'} dD' \quad (2)$$

where D' is an integration variable ranging over the history of the loading program. This equation, analogous to that of linear viscoelasticity [3,4], provides a basic constitutive equation, in which the stress and the strain are linearly related.

Many nuclear grades of graphite exhibit mechanical behaviors which are to various degrees transversely isotropic in nature. Therefore, it is convenient to generalize Eq. (2) to the following multi-axial constitutive equations which are valid for that type of material symmetry:

$$\epsilon_x = J_x * (d\sigma_x - \mu_x d\sigma_y) - \mu_z J_z * d\sigma_z + \alpha_x T + \psi_x(T, D) \quad (3)$$

$$\epsilon_y = J_x * (d\sigma_y - \mu_x d\sigma_x) - \mu_z J_z * d\sigma_z + \alpha_x T + \psi_x(T, D) \quad (4)$$

$$\epsilon_z = J_z * (d\sigma_z - \mu_z d\sigma_x - \mu_z d\sigma_y) + \alpha_z T + \psi_z(T, D) \quad (5)$$

$$\gamma_{xy} = 2(1 + \mu_x) J_x * d\tau_{xy} \quad (6)$$

$$\gamma_{yz} = J_{zx} * d\tau_{yz} \quad (7)$$

$$\gamma_{zx} = J_{zx} * d\tau_{zx} \quad (8)$$

where the z axis is assumed to be the axis of material symmetry and both Poisson ratios μ_x and μ_z to be constants.* The Poisson ratio μ_x is defined as the ratio of induced lateral strain to longitudinal strain for a uniaxial test when both directions lie in the plane of isotropy (x, y). Whereas, μ_z is the ratio of the lateral strain induced in a direction in the plane of isotropy to the longitudinal strain in the direction normal to the isotropic plane. When these Poisson ratios are dose-dependent, two creep functions, in addition to J_x , J_z , and J_{zx} , are required for the stress-strain representation. The notation (*) is used in accordance with the convention for representing a convolution integral,

$$J * d\sigma = \int_0^D J(D - D') \frac{\partial \sigma}{\partial D'} dD \quad (9)$$

The terms αT and $\psi(T, D)$ represent the strains due to thermal expansion and dimensional changes resulting from neutron irradiation, respectively. The neutron dose D will in general be nonuniform throughout a body and plays the same role in these constitutive equations as does the "reduced time" variable used by Muki and Sternberg [7] in the analysis of thermorheologically simple materials.

The generalized plane-strain conditions are defined by the case when the normal strain in a given direction, say the z direction, assumes a constant value ϵ_0 , all derivatives with respect to z vanish,

*Experimental measurements of the strain ratio variables μ_x and μ_z have not been made for graphites during irradiation. However, experimental evidence which tends to support the above assumption has been obtained by Simmons [5]. Strain ratio measurements were made on a number of isotropic reactor grade graphites at room temperature before and after irradiation with no observable change. Experimental strain ratio measurements [6] have also been made at room temperature on a number of anisotropic reactor grade graphites in which a significant amount of plastic deformation occurs. In these experiments the strain ratio values remained reasonably constant over the entire loading range.

and the net resultant force in the z direction vanishes. Under these conditions, Eqs. (7) and (8) identically vanish and Eq. (5) reduces to:

$$J_z * d\sigma_z = \mu_z J_z * (d\sigma_x + d\sigma_y) - \alpha_z T - \psi_z(T, D) + \epsilon_0 \quad (10)$$

Through the use of Eq. (10), the two-dimensional constitutive equations which characterize these generalized plane-strain conditions can be expressed by Eqs. (3), (4), and (6) in the form

$$\begin{aligned} \epsilon_x = (J_x - \mu_z^2 J_z) * d\sigma_x - (\mu_x J_x + \mu_z^2 J_z) * d\sigma_y \\ + (\alpha_x + \mu_z \alpha_z) T + \psi_x + \mu_z \psi_z - \mu_z \epsilon_0 \end{aligned} \quad (11)$$

$$\begin{aligned} \epsilon_y = (J_x - \mu_z^2 J_z) * d\sigma_y - (\mu_x J_x + \mu_z^2 J_z) * d\sigma_x \\ + (\alpha_x + \mu_z \alpha_z) T + \psi_x + \mu_z \psi_z - \mu_z \epsilon_0 \end{aligned} \quad (12)$$

$$\gamma_{xy} = 2(1 + \mu_x) J_x * d\tau_{xy} \quad (13)$$

Thus, for this type of transversely isotropic material, two creep functions J_x and J_z , along with two Poisson ratios μ_x and μ_z , are required for the viscoelastic stress analysis under generalized plane-strain conditions when the isotropic plane coincides with the cross section of the specimen.

For an isotropic graphite, the following simplifications can be made in the generalized plane-strain formulation:

$$\mu_z = \mu_x = \mu$$

$$J_x = J_z = J$$

$$\alpha_x = \alpha_z = \alpha$$

$$\psi_x = \psi_z = \psi$$

and it follows that

$$\epsilon_x = (1 - \mu^2)J * \left(d\sigma_x - \frac{\mu}{1 - \mu} d\sigma_y \right) + (1 + \mu)(\alpha T + \psi) - \mu \epsilon_0 \quad (14)$$

$$\epsilon_y = (1 - \mu^2)J * \left(d\sigma_y - \frac{\mu}{1 - \mu} d\sigma_x \right) + (1 + \mu)(\alpha T + \psi) - \mu \epsilon_0 \quad (15)$$

$$\gamma_{xy} = 2(1 + \mu)J * d\tau_{xy} \quad (16)$$

It is seen from Eqs. (14), (15), and (16) that the viscoelastic stress analysis of an isotropic graphite under these conditions requires the determination of only one creep function $J(D)$, and one Poisson ratio μ . Experimental results [2] which are expressed in Figs. 1 and 2 show that the creep function can be considered as the sum of an elastic contribution, a primary creep term, and a secondary creep term. The primary creep is expressed in terms of the accumulated neutron exposure by

$$J_p(D) = \frac{1}{2E} \left(1 - e^{-A_0 D} \right) \quad (17)$$

and the secondary creep is linearly dependent upon the dose, thus

$$J_s(D) = K(T)D \quad (18)$$

The secondary creep coefficient $K(T)$ is dependent upon the temperature [1], whereas A_0 and E in the primary creep term are constants. The constant A_0 is large in the sense that the second term in the primary creep expression $J_p(D)$ becomes negligible for neutron exposures less than typical current design doses.* In the subsequent analysis, it is assumed that $K(T)$ has a constant value which in practice would correspond to some mean temperature. This assumption is necessary to obtain the governing Eq. (24), given in the next section. The overall creep function, including the elastic part, is then

$$J(D) = \frac{1}{E} + K(T)D + \frac{1}{2E} \left(1 - e^{-A_0 D} \right) \quad (19)$$

This form of the creep function for graphite under irradiation can be represented analogously by a model of springs and dashpots with the spring constants and viscosity coefficients shown in Fig. 4. If A_0 is large, the right dashpot can be removed, leaving two springs and a dashpot connected in series.

FORMULATION AND SOLUTION

In this section the intent is to illustrate the relation between the thermal stress problem in an elastic material and that in a viscoelastic material. Consider an arbitrary two-dimensional cross section where the neutron flux is assumed to be uniform over the

*Typical design doses for the type of reactors considered later in this paper range up to 3×10^{22} nvt.

entire section and the creep function is independent of temperature. As discussed by Sokolnikoff [8], the equations of equilibrium are expressed only in terms of the stress components and not influenced by the constitutive equations; it will be convenient, therefore to introduce the Airy stress function ϕ such that

$$\sigma_x = \frac{\partial^2 \phi}{\partial y^2} \quad (20)$$

$$\sigma_y = \frac{\partial^2 \phi}{\partial x^2} \quad (21)$$

$$\tau_{xy} = - \frac{\partial^2 \phi}{\partial x \partial y} \quad (22)$$

where $\phi = \phi(x, y, D)$ and $D = D(\text{time})$. If there are no body forces, the equations of equilibrium for the generalized plane-strain formulation will be identically satisfied by Eqs. (20), (21), and (22). After substituting these into the equation of compatibility

$$\frac{\partial^2 \epsilon_y}{\partial x^2} + \frac{\partial^2 \epsilon_x}{\partial y^2} = \frac{\partial^2 \tau_{xy}}{\partial x \partial y} \quad (23)$$

the governing equation of ϕ for an isotropic graphite is

$$\int_0^D J(D - D') \frac{\partial}{\partial D'} \nabla^4 \phi \, dD' = - \frac{1}{1 - \mu} \nabla^2 [\psi(D, T) + \alpha T] \quad (24)$$

After inversion, ϕ satisfies

$$\nabla^4 \phi = - \frac{1}{1 - \mu} \int_0^D G(D - D') \frac{\partial}{\partial D'} \left\{ \nabla^2 [\psi(D, T) + \alpha T] \right\} dD' \quad (25)$$

where the relaxation function G is related to the creep function J by

$$\int_0^D G(D - D') \frac{\partial}{\partial D'} J(D') dD' = H(D) \quad (26)$$

and $H(D)$ is the unit step function. Specifically, the relaxation function which corresponds to the creep function given by Eq. (19) is

$$G(D) = \frac{E}{\sqrt{(EK + 1.5A_0)^2 - 4EKA_0}} \left[(k_1 + A_0)e^{k_1 D} - (k_2 + A_0)e^{k_2 D} \right] \quad (27)$$

where

$$k_1 = -0.5(EK + 1.5A_0) + 0.5\sqrt{(EK + 1.5A_0)^2 - 4EKA_0} \quad (28)$$

$$k_2 = -0.5(EK + 1.5A_0) - 0.5\sqrt{(EK + 1.5A_0)^2 - 4EKA_0} \quad (29)$$

and where k_1 and k_2 are seen to be negative. For prescribed boundary traction, the boundary conditions are

$$\frac{\partial \phi}{\partial y} = \int_C T_x \, ds \quad (30)$$

and

$$\frac{\partial \phi}{\partial x} = - \int_C T_y \, ds \quad (31)$$

where T_x and T_y are the x- and y-components of the boundary traction acting on the boundary C of the cross section of the body.

It will be established that if the temperature field is independent of time and if the temperature-dependent neutron-induced dimensional change is given by

$$\psi(D, T) = A_2(T)D^2 + A_1(T)D \quad (32)$$

which is expressed in Fig. 5.

then the stress function ϕ is expressed by

$$\begin{aligned} \phi(x, y, D) = & \phi^a(x, y) + \phi^b(x, y) \frac{G(D)}{G(0)} \\ & + \phi^c(x, y) F_1(D) + \phi^d(x, y) F_2(D) \end{aligned} \quad (33)$$

where ϕ^a , ϕ^b , ϕ^c , and ϕ^d are elastic solutions, corresponding to boundary tractions, thermal expansion, dimensional change $A_1(T)$, and dimensional change $A_2(T)$, respectively, and

$$F_1(D) = \frac{1}{G(0)} \int_0^D G(D - D') dD' \quad (34)$$

and

$$F_2(D) = \frac{1}{G(0)} \int_0^D G(D - D') 2D' dD' \quad (35)$$

In the elastic solutions for the ϕ 's, $E = G(0)$. From this consideration, therefore, the viscoelastic solution for an arbitrary plane-strain problem can be found immediately, provided a nonhomogeneous biharmonic solution is available for calculation of the elastic plane-strain problems with the indicated loadings. In the event that an analytical solution is not available a numerical method could be used.

The above correspondence can be seen from an examination of the solution of Eq. (25) and the boundary conditions Eqs. (30) and (31). The solution for ϕ can be divided into two parts, say ϕ_h and ϕ_p , where the particular solution

$$\phi_p(x, y, D) = -\frac{1}{2\pi(1-\mu)} \int_0^D G(D-D') \frac{\partial}{\partial D'} \left\{ \iint [\psi(D, T) + \alpha T] \log r' d\xi d\eta \right\} dD' \quad (36)$$

in which

$$r' = [(x - \xi)^2 + (y - \eta)^2]^{1/2}$$

and where ξ and η are integration variables ranging over the cross section of the body. Consequently, ϕ_h satisfies

$$\nabla^4 \phi_h = 0 \quad (37)$$

subject to the boundary conditions

$$\frac{\partial \phi_h}{\partial y} = \int T_x ds - \frac{\partial \phi_p}{\partial y} \quad (38)$$

and

$$\frac{\partial \phi_h}{\partial x} = - \int T_y ds - \frac{\partial \phi_p}{\partial x} \quad (39)$$

By substituting the expression given in Eq. (32) for $\psi(D, T)$ into Eq. (36), ϕ_p can be separated as

$$\phi_p(x, y, D) = \frac{G(D)}{G(0)} \phi_p^b(x, y) + F_1(D) \phi_p^c(x, y) + F_2(D) \phi_p^d(x, y) \quad (40)$$

where

$$\phi_p^b(x, y) = -\frac{G(0)}{2\pi(1-\mu)} \iint \alpha T \log r' d\xi d\eta \quad (41)$$

$$\phi_p^c(x, y) = -\frac{G(0)}{2\pi(1-\mu)} \iint A_1(T) \log r' d\xi d\eta \quad (42)$$

$$\phi_p^d(x, y) = -\frac{G(0)}{2\pi(1-\mu)} \iint A_2(T) \log r' d\xi d\eta \quad (43)$$

Moreover, the homogeneous solution ϕ_h is obtained from Eq. (37) and the boundary conditions Eqs. (38) and (39) which are of the same form as for the elastic problem.

The stress function is related to the boundary traction by Eqs. (38) and (39). Hence, the homogeneous solution can be written as

$$\begin{aligned} \phi_h(x, y, D) = & \phi_h^a(x, y) + \frac{G(D)}{G(0)} \phi_h^b(x, y) \\ & + F_1(D) \phi_h^c(x, y) + F_2(D) \phi_h^d(x, y) \end{aligned} \quad (44)$$

where $\phi_h^a(x, y)$ is due to the boundary traction; and $F_1(D)$ and $F_2(D)$ are the same functions used in Eq. (40). Therefore, combining Eqs. (40) and (44) gives

$$\begin{aligned} \phi(x, y, D) = & \phi^a(x, y) + \frac{G(D)}{G(0)} \phi^b(x, y) \\ & + F_1(D) \phi^c(x, y) + F_2(D) \phi^d(x, y) \end{aligned} \quad (45)$$

where $\phi(x, y) = \phi_h(x, y) + \phi_p(x, y)$.

Thus, four elastic solutions ϕ^a , ϕ^b , ϕ^c , and ϕ^d will be enough to describe the thermo-viscoelastic solution for a plane-strain problem with boundary tractions, thermal gradients, and neutron-induced dimensional changes of the form given by Eq. (32) provided that the accumulated neutron dose D is a function only of time and not of position. For problems in which the variation of dose across the cross section is small relative to average, assuming a uniform dose distribution equal to the average yields results which are satisfactory for design purposes.

CONCLUSIONS

The theory of linear viscoelasticity was shown to be suitable to represent the constitutive behavior of graphite bodies exposed to fast-neutron-induced dimensional changes and creep. A theoretical solution was obtained for generalized plane-strain conditions including the effect of boundary tractions, thermal stresses, temperature-dependent dimensional changes resulting from fast-neutron irradiation, and a temperature-dependent creep function.

When the fast neutron flux is constant over the cross section of the body, an important consequence of the present formulation is that the viscoelastic solution for any two-dimensional problem, subject to the above conditions, can be obtained from the sum of four associated elastic solutions which are multiplied by appropriate functions of the neutron dose. The elastic solutions require, at most, the use of a numerical method for calculating elastic thermal stresses.

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Fig. 1. Creep of AGOT Graphite Under Irradiation.

Fig. 2. Creep of Graphite Under Irradiation.

Fig. 3. Linear Superposition for a Viscoelastic Material.

Fig. 4. Viscoelastic Model Representation for Irradiated Graphite.

Fig. 5. Neutron-Induced Dimensional Changes in Graphite at Various Temperatures.

STRUCTURAL ANALYSIS — NUCLEAR

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VISCOELASTIC RESPONSE OF GRAPHITIC MATERIALS IN IRRADIATION ENVIRONMENTS*

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A numerical method is presented for the stress analysis of reactor graphitic components under conditions of plane stress, plane strain, generalized plane strain or axisymmetry. In the analysis, both the creep deformation and the irradiation-induced strains are the major concern. The mechanical response of the material is assumed to be linear viscoelastic and transversely isotropic. The constitutive relations are formulated in hereditary integral form, and the dependence of the material properties on temperature and neutron flux are considered. To illustrate the method, a numerical example on the analysis of a fuel element is presented.

1. Introduction

Many reactor components (e.g., moderator, reflector, fuel element, fuel matrix, etc.) are made of graphite materials and graphite composites. Among various structural design problems, creep deformation and irradiation induced strains are important considerations. In some cases, because of possible stress relief, creep may be beneficial, especially in the stress concentration regions. However, in other cases it introduces undesirable geometry distortions or creep rupture. Furthermore, irradiation-induced strains in a solid are equivalent to thermal loading and will give rise to stresses and strains which may be critical in design. Therefore, if one is to carry out a realistic stress analysis, it is necessary to consider the effect of irradiation induced creep and dimensional changes.

In this paper, the mechanical response of graphite is assumed to be linear viscoelastic and the constitutive relations are formulated in hereditary integral form. The material properties are considered to be temperature as well as neutron flux dependent. Irradiation-induced dimensional changes are treated as equivalent thermal strain. The general viscoelectric problem is formulated and solution techniques are briefly discussed. It is shown that, in order to solve a general class of problems, a numerical approach

which utilizes the finite element method is most effective. A computer program was consequently developed for the analysis of plane strain, generalized plane strain and axisymmetric problems. For illustration, a numerical example on the analysis of a fuel element is presented. The fuel element is deformed under variable temperatures and irradiation-induced strains with neutron flux up to 5×10^{21} nvt. Results in the form of stress contours and stress histories are presented.

2. Constitutive equations

Most graphitic components in a reactor are fabricated by extrusion, causing the grains to be oriented preferentially with respect to the direction of extrusion. This orientation causes the mechanical properties such as the coefficient of thermal expansion, the thermal conductivity and the elastic and inelastic moduli of a graphite body to be anisotropic. However, in the plane normal to the direction of extrusion the material behavior is still isotropic. Thus, if one of the three orthogonal reference coordinates coincides with the extrusion axis, the graphite material is transversely isotropic. Furthermore, graphite undergoes temperature dependent creep

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deformations and dimensional changes when subjected to fast neutron flux [1, 2].

Within the scope of small deformation theory, it is assumed that the creep response of graphite is linearly proportional to the applied stresses. For example, consider a uniaxial creep experiment in which the graphite specimen is deformed by a constant stress σ_0 under a uniform temperature T . The linearity assumption implies that the measured creep strain ϵ (excluding the thermal and irradiation strains) can be expressed by

$$\epsilon(\gamma) = J(\gamma, T) \sigma_0, \quad (1)$$

where γ is the fast neutron dose; J , a uniaxial creep function having the unit in./in./psi. The assumption also implies that if the neutron flux field is sustained, there will be complete delayed creep recovery when the stresses are removed.

If the material is subjected to a variable stress history, the linear superposition principle [4] extends eq. (1) to

$$\epsilon(\gamma) = \int_0^\gamma J(\gamma - \gamma', T) (d/d\gamma') \sigma(\gamma') d\gamma', \quad (2)$$

where the lower integration limit 0^- provides for the elastic response at $\gamma = 0$.

Eq. (2) is valid only for a uniaxial stress state and a homogeneous neutron flux field. Since we expect to deal with multiaxial stress states and a spatially variable flux, it would be more convenient to use the time as an independent variable instead of the neutron flux. For this, the strain-stress relation is written as*

$$\epsilon(t) = \int_0^t J(t - t', T, \phi) (d/dt') \sigma(t') dt', \quad (3)$$

where ϕ denotes the intensity of the neutron flux field such that

$$\gamma = \gamma(x_i, t) = \phi(x_i) t \quad (4)$$

* Note that the function $J(t, T, \phi)$ is different from $J(\gamma, T)$. However, for simplicity we use the same notation.

and t represents either the real time or any normalized reference time.

The inverse (or stress-strain) relation of (3) is given by

$$\sigma(t) = \int_0^t G(t - t', T, \phi) (d/dt') \epsilon(t') dt', \quad (5)$$

where G is the relaxation modulus which is related to the creep compliance J under constant temperature and neutron dose as follows:

$$h(t) = \int_0^t J(t - t', T, \phi) (d/dt') G(t') dt', \quad (6)$$

where $h(t)$ is the unit step-function. It is clearly seen in (6) that if one of the material functions, J or G , is given, the other one can be determined by solving the integral equation.

If the graphite body is deformed under a two- or three-dimensional state of stress, constitutive equations for transversely isotropic materials have to be formulated. For this purpose, let us consider a rectangular Cartesian coordinate system x_i , $i = 1, 2, 3$, of which the x_3 axis is pointed along the extrusion direction. In this reference frame, the material is isotropic in the (x_1, x_2) plane and orthotropic with respect to 1, 3 and 2, 3 directions. To define the mechanical state in graphite, let σ_{ij} , ϵ_{ij} be the stress and strain tensors, respectively. The Onsager's reciprocity relations in thermodynamics requires that the viscoelastic behavior of transversely isotropic materials is characterized by five independent material functions [5].

In view of the uniaxial relationship, the three-dimensional strain-stress law may be expressed in matrix notation as follows:

$$\{\epsilon(t)\} = \int_0^t [C(t - t', T, \phi)] (d/dt') \{\sigma(t')\} dt' + \{\theta(t)\} + \{\epsilon^o(t)\}, \quad (7)$$

where C represents the matrix of creep compliances; θ and ϵ^o are the thermal and irradiation strain

vectors, respectively. Since the stress and the strain are generally functions of space x_i as well as time t , partial derivatives are employed in (7).

Similarly, the stress-strain relations are given by

$$\{\sigma(t)\} = \int_0^t [R(t-t', T, \phi)] (\partial/\partial t') \times (\{\epsilon(t')\} - \{\theta(t')\} - \{\epsilon^o(t')\}) dt', \quad (8)$$

where R represents the matrix of relaxation moduli. Further details of the constitutive relations are included in Appendix A.

3. Variational solution

The classical approach to the solution of linear quasi-static boundary value problems is employed by transform techniques. In this respect, Laplace transform is generally applied to the field and equilibrium equations thereby reducing the equations for the viscoelastic problem to a form similar to the elastic problem. This direct analogy lead to the correspondence principle in linear viscoelasticity [4], which states that: if the solution of an elastic problem exists, the solution of the associated viscoelastic problem is obtained by replacing the moduli in the elastic solution by the equivalent viscoelastic moduli in the transformed space. Of course, the final solution is the Laplace inverse of the transformed expression.

In the case at hand where inhomogeneities are introduced due to non-uniform temperature and neutron flux fields, the correspondence principle is very difficult to apply. The major difficulties are mainly encountered in the process of Laplace inversion, and only in oversimplified cases which are of no practical importance can a solution be obtained.

In order to treat a general class of problems, a numerical approach based on the finite element variational method is adopted. In this approach, the field equations are derived by the aid of Laplace transform for each element where the neutron flux and temperature fields are assumed to be locally uniform. In this manner, the difficulties arising from inhomogeneous distributions are removed. It should

be pointed out, however, that temperature and neutron flux fields that are globally non-uniform are treated as piecewise uniform fields. Once the equilibrium equations are derived, in the time domain, for each element they are merged to form the complete equilibrium equations of the total solid. Prior to this development, however, the appropriate variational principle is discussed.

3.1. Variational principle

In elastostatics, an equivalent statement of the mathematical problem may be made in terms of the minimization of a functional. Instead of dealing with the partial differential field equations, formulation of the problem is arrived at by a variational process on an integral involving the potential energy of the elastic body. This procedure has been defined as the theorem of minimum potential energy [6]. A similar approach can be made for a viscoelastic body in the Laplace transformed space [7].

Under uniform temperature and neutron flux fields, the creep compliances C in (7) and the relaxation moduli R in (8) may be replaced respectively by

$$\begin{aligned} C &= C(t-t'), \\ R &= R(t-t'). \end{aligned} \quad (9)$$

Applying Laplace transform on (7) and (8), we obtain

$$\{\bar{\epsilon}(\xi)\} = [\bar{C}(\xi)] \xi \{\bar{\sigma}(\xi)\} + \{\bar{\theta}(\xi)\} - \{\bar{\epsilon}^o(\xi)\}, \quad (10)$$

$$\{\bar{\sigma}(\xi)\} = [R(\xi)] \xi (\{\bar{\epsilon}(\xi)\} - \{\bar{\theta}(\xi)\} - \{\bar{\epsilon}^o(\xi)\}), \quad (11)$$

where the Laplace transform of a function f is defined by

$$\bar{f}(\xi) = \int_0^\infty e^{-\xi t} f(t) dt; \quad (12)$$

ξ is the transform parameter. Now consider a viscoelastic body occupying a domain V with boundary Σ under the influence of either a steady-state or a slowly varying temperature field T . The solid is subjected to body forces f_i in V and surface tractions P_i on part

of the boundary Σ_σ . Then, in the transformed space among all the displacements u_i satisfying the geometric boundary condition, those which make the potential energy $\bar{\pi}$ an absolute minimum satisfy the equilibrium equations and stress boundary condition. This may be stated mathematically,

$$\delta \bar{\pi} = 0, \quad (13)$$

where $\bar{\pi}$ is defined as the associated potential energy for a viscoelastic body, and

$$\begin{aligned} \bar{\pi} = & \int_V \left(\frac{1}{2} ([\bar{R}] \xi^2 \{\bar{\epsilon}\})^* \{\bar{\epsilon}\} \right. \\ & \left. - [\bar{R}] \xi^2 (\{\bar{\theta}\} + \{\bar{\epsilon}^o\})^* \{\bar{\epsilon}\} \right) dv \\ & - \int_V \{\bar{f}\}^* \xi \{\bar{U}\} dv - \int_{\Sigma_\sigma} \{\bar{P}\}^* \xi \{\bar{U}\} d\Sigma. \end{aligned} \quad (14)$$

In (14) $\{\}^*$ denotes the transpose of the vector. The Laplace inverse of (14) corresponds to the second variational principle given in [8].

3.2. Finite element formulation

With the appropriate variational principle, eqs. (13) and (14), in hand, we will now outline a numerical solution procedure based on the finite element method. We require that the linearized strain-displacement relations

$$\epsilon_{ij} = \frac{1}{2} (u_{i,j} + u_{j,i}) \quad (15)$$

remain valid. In addition, the stresses and displacements must satisfy the appropriate conditions on the boundary. The stress boundary condition is given by

$$\sigma_{ij} n_j = P_i \quad (16)$$

on a portion of the boundary Σ_σ where P_i and n_j are components of the traction vector and unit outward normal respectively. The displacements satisfy

$$u_i = U_i \quad (17)$$

on the remaining surface Σ_u where U_i are known displacements.

Let the solid be divided into a number of small closed regions ΔR_m , $m = 1, 2, \dots, M$. In each region the displacement functions u_i are approximated in terms of coordinate functions $\phi_{ij}(x)$ and the generalized coordinates q_j as follows:

$$u_i(x,t) = \phi_{ij}(x) q_j(t), \quad (18)$$

where x represents the position vector with components x_1, x_2, x_3 . If the number of displacement components is N , and the number of nodal points of the element is R , then the index i ranges from 1 to N , and j ranges from 1 to $N \times R$. In matrix notation, eq. (18) can be written as

$$\{u(x)\} = [\Phi(x)] \{q(t)\}. \quad (19)$$

Substituting (19) into the strain-displacement relations (15), the strains are related to the generalized coordinates $q_j(t)$ by

$$\{\epsilon\} = [\psi] \{q\}. \quad (20)$$

The generalized coordinates $q_j(t)$ are in turn related to the nodal displacements $u_j(t)$ at the nodal points by a linear transformation derived from (19), i.e.,

$$\{q(t)\} = [A] \{v(t)\}. \quad (21)$$

Applying Laplace transform to eqs. (19) thru (21) and substituting into (13) and (14) yields

$$\begin{aligned} & \delta \left(\sum_{i=1}^M \int_{\Delta R_i} [\bar{R}] \xi \left(\frac{1}{2} [\psi] [A] \{\bar{v}\} - \{\bar{\theta}\} - \{\bar{\epsilon}^o\} \right)^* \right. \\ & \quad \times \xi ([\psi] [A] \{\bar{v}\}) dv \\ & \quad - \sum_{i=1}^M \int_{\Delta R_i} \{\bar{f}\}^* \xi [\Phi] [A] \{\bar{v}\} dv \\ & \quad \left. - \sum_{i=1}^S \int_{\Delta \Sigma_i} \{\bar{P}\}^* \xi [\phi] [A] \{\bar{v}\} d\Sigma \right) = 0. \end{aligned} \quad (22)$$

where M is the total number of elements that comprise the continuum, S is the number of segments

along the traction boundary. The variation of (22) leads to the equations

$$\frac{\partial(\quad)}{\partial(\xi \bar{v}_i)} = 0 \quad (23)$$

for $i = 1, 2, \dots$, number of unknown displacements.

Explicitly, eqs. (23) in matrix form are

$$[K] \xi \{\bar{v}\} = \{\bar{F}\}. \quad (24)$$

From the Laplace inverse of (24), we obtain

$$\int_0^t [K(t-t')] (d/dt') \{u(t')\} dt' = \{F(t)\}. \quad (25)$$

Eq. (25), which is known as the equilibrium condition for the discretized viscoelastic solid, represents a set of linear integral-differential equations. These equations can be solved by numerical integration procedures [11].

Although the above algorithm is valid for general three-dimensional state of stress and deformation, a computer program has been developed for solving two-dimensional problems; i.e., problems with plane strain, generalized plane strain or axisymmetric deformations. These are defined in Appendix B.

4. Numerical example

A fuel element subjected to variable temperature field and neutron exposure to 5×10^{21} nvt was analyzed. The analysis was carried out for the central portion of the fuel block where the neutron intensity and temperature are most severe. Because of the symmetry in geometry and temperature field, only a sector of the fuel element was considered, as shown in fig. 1. A total of 222 elements and 132 nodal points were used in the finite element idealization as shown in fig. 2. In the analysis, a generalized plane strain was assumed to prevail. The creep compliance is assumed to be a linear function of time as given in eq. (A16) and the creep coefficient used was taken from ref. [3]. The dimensional changes as functions of temperature and neutron dose are obtained from experimental measurements. The

Fig. 1. A typical fuel element.

Fig. 2. Finite element grid.

Fig. 3. Axial stresses in psi.

maximum temperature difference in the sector is about 70°F from one end to the other and the average temperature varies from 240°F to 1550°F throughout the irradiation history. The reactor was shut down for several times during its operation.

The elastic moduli in the axial direction and the isotropic plane were chosen to be 1.86×10^6 psi and 0.66×10^6 psi, respectively. The Poisson ratio used is 0.2.

Results are presented in figs. 3–6. Maximum values of all the stress components occurred at the neutron dose $\gamma = 2.4 \times 10^{21}$ nvt. In fig. 3, the axial stress contours are given for two different neutron exposures. It is seen that stress relaxation occurred between $\gamma = 2.4$ and $\gamma = 5 \times 10^{21}$ nvt where the patterns of stress contours changes significantly. This indicates that due to the creep phenomenon, redistribution of stresses has occurred. Similar stress contours for the maximum principal stress in the

Fig. 4. Maximum principal stress in isotropic plane.

isotropic plane are also shown in fig. 4. The in-plane stress is considerably lower than the axial stress due to the deformation condition of generalized plane strain.

In figs. 5 and 6, the axial stress history for points near the fuel hole and coolant are given respectively. For example, in fig. 5 the stress has an initial value of -50 psi due to steady-state temperature gradient. When the reactor is in operation, the stress increases to tension due to the effect of irradiation-induced dimensional changes. At $\gamma = 2.4 \times 10^{21}$ nvt, the axial stress decreases indicating dominance of the stress relaxation phenomenon. Because of the reactor shut down at various stages, a number of spikes are shown along the curve. In fig. 6, the axial stress near the coolant hole shows similar behavior except in the reversed trend.

Fig. 5. Axial stress history near the fuel hole.

Fig. 6. Axial stress history near the coolant hole.

5. Conclusion

A numerical method has been presented for the analysis of graphite structures under irradiation condition. A computer program was written to analyze plane strain, generalized plane strain and axisymmetric problems. Because of the use of the finite element method, non-uniform temperature and neutron flux fields, irregular geometries and general boundary conditions can be handled. In addition, this solution method can be applied not only to the graphite structures, but also to other materials (uranium fuel, steel, etc.) exhibiting similar behavior under irradiation.

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Appendix A. Constitutive equations in engineering parameters

For linear viscoelastic and transversely isotropic

material, the strain-stress relations are given by

$$\epsilon(t) = \int_0^t [C(t-t', T, \phi)] (\partial/\partial t') \{\sigma(t')\} dt' + \{\theta(t)\} + \{\epsilon^\circ(t, T)\}, \quad (A1)$$

where the strain and stress vectors are defined by

$$\begin{aligned} \{\epsilon\}^* &= (\epsilon_{11}, \epsilon_{22}, \epsilon_{33}, \epsilon_{12}, \epsilon_{23}, \epsilon_{13}), \\ \{\sigma\}^* &= (\sigma_{11}, \sigma_{22}, \sigma_{33}, \sigma_{12}, \sigma_{23}, \sigma_{13}). \end{aligned} \quad (A2)$$

The thermal strain

$$\{\theta\} = \int_{T_0}^T \{\alpha(T')\} dT', \quad (A3)$$

with

$$\{\alpha\}^* = (\alpha_{11}, \alpha_{11}, \alpha_{33}, 0, 0, 0), \quad (A4)$$

and the irradiation strains

$$\{\epsilon^\circ\}^* = (\epsilon_{11}^\circ, \epsilon_{11}^\circ, \epsilon_{33}^\circ, 0, 0, 0). \quad (A5)$$

The creep-compliance matrix C consists of five independent material functions such that

$$[C] = \begin{bmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{13} & 0 & 0 & 0 \\ C_{13} & C_{13} & C_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{2}(C_{11}-C_{12}) \end{bmatrix} \quad (A6)$$

where $C_{ij} = C_{ij}(t, T, \phi)$.

For engineering problems, it is sometimes preferable to express the material functions in (A6) in terms of uniaxial moduli and Poisson's ratios. This may be achieved by performing the uniaxial creep tests so that

$$\begin{aligned} C_{11} &= J_1, \\ C_{12} &= -\nu_1 J_1, \\ C_{13} &= -\nu_{31} J_1 = -\nu_{13} J_3, \\ C_{33} &= J_3, \\ C_{44} &= J_4, \end{aligned} \quad (A7)$$

where J_1 and J_3 are the extensional creep compliances in the isotropic plane and the x_3 direction, respectively; J_4 , the shear compliance in the (x_3x_1) , (x_3x_2) planes; ν_1 , the Poisson's ratio in the isotropic plane; ν_{13} , the Poisson's ratio in the x_3 direction due to the stress applied in the isotropic plane, and ν_{31} is similarly defined.

In general, the creep compliances and the Poisson's ratios are functions of time, neutron intensity and temperature.

It is seen in (A7) that an extensive creep test program is required to characterize the material functions. In some cases, it may be reasonable to assume that the Poisson ratios are merely constants and the creep compliances are all proportional to a single function, i.e.,

$$\begin{aligned} J_1 &= \kappa_1 J(t, T, \phi), \\ J_3 &= \kappa_3 J(t, T, \phi), \\ J_4 &= \kappa_4 J(t, T, \phi), \end{aligned} \quad (A8)$$

where κ_1 , κ_3 and κ_4 are constants. The assumption on constant Poisson ratios is justified by the fact that the variation in Poisson ratio of irradiated graphite is small [12] and the effect on stress distribution would not be significant.

In view of (A7) and (A8), we rewrite the strain-stress relations

$$\begin{aligned} \{\epsilon(t)\} &= [L] \int_0^t J(t-t', T, \phi) (\partial/\partial t') \{\sigma(t')\} dt' \\ &+ \{\theta(t)\} + \{\epsilon^\circ(t, T)\}, \end{aligned} \quad (A9)$$

where L is a constant matrix,

$$[L] = \begin{bmatrix} \kappa_1 & -\nu_1 \kappa_1 & -\nu_{31} \kappa_1 & 0 & 0 & 0 \\ -\nu_1 \kappa_1 & \kappa_1 & -\nu_{31} \kappa_1 & 0 & 0 & 0 \\ -\nu_{13} \kappa_3 & \nu_{13} \kappa_3 & \kappa_3 & 0 & 0 & 0 \\ 0 & 0 & 0 & \kappa_4 & 0 & 0 \\ 0 & 0 & 0 & 0 & \kappa_4 & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{2}(1+\nu_1)\kappa_1 \end{bmatrix} \quad (\text{A10})$$

with $\nu_{31}\kappa_1 = \nu_{13}\kappa_3$.

Inverting (A9), the stress-strain relations are found to be the following:

$$\{\sigma(t)\} = [H] \int_0^t G(t-t', T, \phi) (\partial/\partial t') \times (\{\epsilon(t')\} - \{\theta(t')\} - \{\epsilon^\circ(t', T)\}) dt', \quad (\text{A11})$$

where G is the relaxation modulus; H is the inverse of the L -matrix and has the following explicit expression:

$$[H] = \begin{bmatrix} H_{11} & H_{12} & H_{13} & 0 & 0 \\ H_{12} & H_{11} & H_{13} & 0 & 0 \\ H_{13} & H_{13} & H_{33} & 0 & 0 \\ 0 & 0 & H_{44} & 0 & 0 \\ 0 & 0 & 0 & H_{44} & 0 \\ 0 & 0 & 0 & 0 & H_{66} \end{bmatrix} \quad (\text{A12})$$

$$H_{11} = a/(a^2 - b^2),$$

$$H_{12} = b/(a^2 - b^2),$$

$$H_{13} = \nu_{13}/(a - b),$$

$$H_{33} = (1/\kappa_3) + [2\nu_{13}^2/(a - b)],$$

$$H_{44} = 1/\kappa_4,$$

$$H_{66} = 1/[2(1 + \nu_1)\kappa_1],$$

$$a = \kappa_1 - \nu_{13}^2 \kappa_3, \quad (\text{A13})$$

$$b = \nu_1 \kappa_1 + \nu_{13}^2 \kappa_3. \quad (\text{A13}')$$

For the purpose of engineering analysis, it is sometimes more desirable to represent the creep or relaxation data by mathematical expressions. To this end, we assume that the creep and relaxation functions have the following forms:

$$J(t, T, \phi) = \frac{1}{E(T)} + \eta(T) \phi t + \sum_{k=1}^m \{c_k(T) (1 - \exp[-\xi_k(T) \phi t])\}, \quad (\text{A14})$$

and

$$G(t, T, \phi) = \sum_{k=1}^n g_k(T) \exp[-\tau_k(T) t] + g_\infty(T), \quad (\text{A15})$$

where E is Young's modulus, η , C_k , g_k , g_∞ are parameters determined from fitting the material data.

If the creep compliance can be adequately represented by a linear function of time,

$$J(t, T, \phi) = \frac{1}{E(T)} + \eta(T) \phi t, \quad (\text{A16})$$

then from eq. (6), the relaxation modulus must have the form

$$G(t, T, \phi) = E(T) \exp[-E\eta\phi t], \quad (\text{A17})$$

where $\eta(T)$ is usually called the creep coefficient.

Appendix B. Plane deformation states

B1. Plane strain and generalized plane strain deformations

The non-vanishing stress and strain components are given by

$$\{\sigma\} = \begin{Bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{12} \end{Bmatrix}, \quad \{\epsilon\} = \begin{Bmatrix} \epsilon_{11} \\ \epsilon_{22} \\ \epsilon_{33} \\ \epsilon_{12} \end{Bmatrix}, \quad (\text{B1})$$

and

$$\{\theta\} = \begin{Bmatrix} \theta_{11} \\ \theta_{11} \\ \theta_{33} \\ 0 \end{Bmatrix}, \quad \{\epsilon^\circ\} = \begin{Bmatrix} \epsilon^\circ_{11} \\ \epsilon^\circ_{11} \\ \epsilon^\circ_{33} \\ 0 \end{Bmatrix}, \quad (\text{B2})$$

$$[H] = \begin{bmatrix} H_{11} & H_{12} & H_{13} & 0 \\ H_{12} & H_{11} & H_{13} & 0 \\ H_{13} & H_{13} & H_{33} & 0 \\ 0 & 0 & 0 & H_{66} \end{bmatrix}. \quad (\text{B3})$$

In the strain vector, the plane strain deformation requires

$$\epsilon_{33}(x,t) = 0. \quad (\text{B4})$$

Whereas in the case of generalized plane strain, it requires

$$\epsilon_{33} = c(t), \quad (\text{B5})$$

where c is determined from the condition that the total axial force in the isotropic plane, i.e., (x_1, x_2) plane, must vanish,

$$\int_A \sigma_{33}(x,t) dA = 0. \quad (\text{B6})$$

B2. Axisymmetric deformation

The state of stress and strain is defined by

$$\{\sigma\} = \begin{Bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{13} \end{Bmatrix}, \quad \{\epsilon\} = \begin{Bmatrix} \epsilon_{11} \\ \epsilon_{22} \\ \epsilon_{33} \\ \epsilon_{13} \end{Bmatrix}. \quad (\text{B7})$$

The thermal strain $\{\theta\}$, irradiation strain $\{\epsilon^\circ\}$, and the material matrix $[H]$ are the same as given by (B2) and (B3) except that the element H_{66} in (B3) is replaced by H_{44} according to (A13).

✓ ANALYSIS OF SPHERICAL COATINGS ON NUCLEAR FUEL PARTICLES

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ABSTRACT

Development of coated-particle fuels for high-temperature gas-cooled reactors has prompted the formulation of mathematical models which permit estimation of the operating life of pyrolytic carbon- and/or silicon carbide-coated nuclear fuel microspheres. Although such models are of limited utility in a priori failure predictions, they are of significant value in the planning and interpretation of irradiation tests of such fuels, and in extrapolating test data to new operating conditions.

These models vary in complexity and generality, but commonly involve analysis of the particle coating as a multi-layer spherical pressure vessel, subjected to loading from internal fission-gas pressure, fuel swelling, and the shrinkage and anisotropic distortion of coating materials resulting from fast-neutron damage. Failure is predicted to occur when some selected function of the coating stress and/or strain exceeds a specified minimum value.

Such an analysis is presented for two-layer spherical pyrolytic-carbon shells surrounding microspheres of $(U,Th)C_2$ or $(U,Th)O_2$. These coatings may be anisotropic and exhibit significant creep under fast-neutron irradiation, and an iterative numerical technique is employed in determining the distribution of coating stresses and strains. Results are found to be rather sensitive to the magnitude of radiation-induced dimensional changes and creep assumed for the coatings. However, comparisons between model predictions and the results of coated-particle irradiation and annealing experiments have been generally encouraging.

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INTRODUCTION

High-temperature gas-cooled nuclear reactors should have a unique interest for the stress analyst, for their currently-proposed designs incorporate some of the largest, and certainly the smallest, pressure vessels devised by man. The large examples are the prestressed concrete reactor vessels, which provide both nuclear shielding and high-pressure containment for the helium coolant. For a 1000-megawatt electrical reactor, such a vessel might be a vertical 90 ft.-diameter cylinder, well over 100 ft. tall, with 10-15 ft.-thick walls. At the small end of the size spectrum are the pyrolytic carbon coatings surrounding fuel microspheres ((U,Th)C₂ or (U,Th)O₂). It is with these tiny pressure vessels--usually less than one millimeter in outside diameter, with wall thickness of the order of 0.1 millimeter--that this discussion will be concerned.

A typical coated fuel particle is illustrated schematically in Figure 1. Fuel rods consisting of these particles, dispersed in a carbon binder matrix, are located in holes drilled in a machined graphite block. Other holes in the block provide flow channels for the coolant helium. Such a fuel element is to be used in the prototype Ft. St. Vrain HTGR designed by Gulf General Atomic for the Public Service Company of Colorado, and now under construction¹.

The fuel particle itself contains a spherical kernel of uranium or uranium-thorium dicarbide. The layer surrounding the kernel is a low-density pyrolytic carbon "buffer" whose function is to provide free volume to accommodate fuel swelling and gaseous fission products released from the kernel. The outer high-density pyrolytic carbon layers are largely impervious to gaseous fission products and form the structural portion of the tiny pressure vessel. The particle illustrated incorporates a layer of silicon carbide to act as a barrier to diffusion of metallic fission products, especially strontium and barium.

In designing coated particles for a given reactor application, it would be desirable to be able to predict quantitatively the influence of a coated particle's configuration and material properties on its expected life to failure in a given irradiation environment. To facilitate such predictions, mathematical models describing coated-particle irradiation behavior have been developed²⁻¹¹. Although uncertainties in individual particle dimensions and material properties limit the utility of such models for reliable a priori predictions of coated particle failure, they do provide a rational means for interpolation between limited irradiation test results and for assessing the relative influence of various coated-particle design variables on expected irradiation performance.

MATHEMATICAL MODELS

The central feature of mathematical models of coated-particle irradiation behavior proposed to date is a stress analysis of the particle coating, idealized as a thick-walled, spherical, single-or multi-layer pressure vessel, as shown in Figure 1. Development and refinement of coated-particle mathematical models has been stimulated by increasing knowledge of the irradiation behavior of pyrolytic-carbon coating materials, and the development of complex multi-layered coating designs. Early models were based on the assumption that the single outer coating layer was isotropic and elastic, with the only loading arising from internal fission-gas pressure. This approach was used successfully by Schwartz⁵ to correlate the results of low-fast fluence, high-burnup irradiation tests; but it could not represent the effects of anisotropic distortion resulting from fast-neutron damage in the pyrolytic carbon, and our initial work in this area at ORNL⁷ took such effects into account. The publication of results by Price and Bokros in 1966, showing that pyrolytic carbon creeps

significantly under fast-neutron irradiation¹², required major model modifications to represent this effect^{6,7}. The development of multi-layered coatings utilizing silicon-carbide barriers and/or different grades of pyrolytic carbon led Kaas^{8,9} of Gulf General Atomic and Walther¹⁰ and Novario¹¹ of the Dragon Project in Europe to generalize our treatment to describe the stress state in multi-layer coatings, including the effects of layer separation and fracture of individual layers on the coating stress history. Computer programs to implement these calculations are available for most of the models in current use.

To illustrate the calculations employed in coated-particle mathematical models, we shall outline the development for a two-layer coating incorporating an inner buffer layer and a single outer pyrolytic-carbon structural layer. Tangential tensile stresses are set up in this outer coating as a result of the fission-gas pressure acting against its inner surface and, if the coating is anisotropic, from fast-neutron damage. The deformation tendencies leading to these stresses are illustrated in Figure 2. In these drawings the initial shape is indicated by solid lines and the deformed shape by shaded areas. In the upper left-hand figure is shown the type of deformation produced by internal pressure from fission gas or fuel kernel swelling. The bottom figure shows the deformation of a low-density isotropic coating that shrinks uniformly under fast-neutron irradiation onto a rigid substrate, such as a layer of silicon carbide. In both these cases the tangential stress system produced is tensile throughout. In the upper right-hand figure we see the type of deformation produced by the anisotropic dimensional changes in pyrolytic carbon, caused by fast-neutron damage (which promotes radial expansion and tangential contraction), or by thermal expansion (which is greater in the radial than in the tangential direction). In these cases, tangential tension occurs near the outer surface and compression near the inner surface.

In a real coated fuel particle, undergoing irradiation, a complex combination of these deformation tendencies may occur; and the interaction between fission-gas pressure, coating stress, and coating displacement, necessitates a trial-and-error solution for the stress distribution, even when elastic behavior of the coating is assumed. The computational difficulties are significantly increased by the inclusion of creep, since this introduces nonlinearity and time dependence into the equations governing stress and strain in the spherical coatings, and makes it difficult to obtain explicit solutions for the stress distribution. The approach we have employed for the calculation of coating stress distributions in the presence of creep is based on the iterative numerical procedure described by Mendelson, Hirschberg, and Manson¹³. In this technique, equations giving the coating stress distributions in terms of initially unknown creep displacements are employed, together with the creep rate-stress relations, in a trial-and-error fashion to obtain the distributions of average coating stress and displacement over a small specified time interval. The procedure is repeated over successive time intervals to provide a coating stress-distribution history applicable for given coated-particle dimensions, properties, and irradiation environment.

The relations used in the stress analysis of the outer coating are shown in the next series of figures. Figure 3 lists the equation of equilibrium for a thick-walled spherical shell, loaded symmetrically, together with the boundary conditions we have employed--fission-gas pressure exerted against the inner surface and no significant restraint at the outer surface.

In Figure 4 are shown the relations between coating displacements and stresses for a spherical shell of anisotropic material whose symmetry axes coincide with the radial direction. The "g" functions represent fractional displacements caused by factors other than elastic response to stress, e.g., thermal expansion, fast-neutron induced shrinkage or growth, and creep. In the

absence of creep the g 's may usually be taken as independent of radial position. However, when creep is taken into account as indicated in Figure 5, the g 's contain time-integrated strain rates, which will show strong radial variations, since they depend on the stresses which are, themselves, radially dependent. Combination of these relations and solution of the resulting differential equations lead to solutions for the radial and tangential stress distributions as illustrated in Figures 6 and 7. It is important to note that these stress distribution relations only seem to be explicit, since as shown in Figure 8, the "I" integrals contain the g 's, which include time integrals of the creep strain rates, which, in turn, depend on the stresses. Hence, stress distributions are calculated in an iterative fashion.

To obtain relations between the stress components and creep-strain rate components, we have used relations based on Hill's theory of anisotropic yielding¹⁴ together with ideas supplied by C. R. Kennedy based on his experience with graphite⁷. These are shown in Figure 9, which illustrates relations between the effective creep strain rate and the strain rate components, and between the effective creep stress and creep strain rate. This latter is a simple proportional relation of the type assumed by Price and Bokros¹² in treating their pyrocarbon creep results. For these linear creep relations Stevens¹⁵ has derived an explicit solution for stress, eliminating the need for the creep portion of the trial and error procedure, increasing calculation speed, and improving accuracy.

The calculation procedure may be summarized by reference to Figure 10. (1) First a time (or fluence) interval is chosen, small enough that the use of average stress values over the interval will not introduce serious error. (2) Next, average values for pressure and creep-strain increments are assumed over the interval, and these are used to calculate a first approximation to the stress distributions (top-center block). (3) These stresses are then used to correct the creep strain increments (lower left-hand block), and the procedure is repeated until a converged set of stresses, strains, and creep-strain increments is obtained for the assumed pressure. (4) The pressure estimate is then corrected through the right-hand calculation loop shown in Figure 10. A numerical technique to speed convergence is employed here, but for each new trial pressure, trial-and-error calculations are performed through the left-hand loop to obtain stresses and creep strain increments satisfying the equations for the given pressure. (5) When convergence finally occurs in both loops, a new time interval is selected and the entire procedure repeated. Except for the first interval, starting guesses for pressure and creep strain increments are usually based on converged values from the preceding interval; this accelerates over-all convergence considerably.

These calculations continue over successive time intervals until some criterion for termination is met: this is normally the attainment of a given total fluence or time, or the exceeding of a specified stress state at some point in the coating.

Before moving on to some illustrative results, an additional point should be noted; namely that this calculation procedure is suitable for use with almost any conceivable creep relation. In our calculations here, we have assumed extremely simple relations, with creep strain rates proportional to the product of the fast-neutron flux and maximum shear stress at each point in the coating. However, since these creep strain rates are calculated in separate subroutines, it would be a simple matter to substitute more complicated creep relations should they be justified by future experimental measurements.

COMPARISONS BETWEEN MODEL PREDICTIONS AND EXPERIMENT

At low fast-neutron exposures, good agreement has been obtained between model predictions and observed coated-particle performance over a wide range of burnups. Comparison of predicted and observed results is shown in Figure 11 for a set of irradiation tests on pyrolytic-carbon-coated UO_2 and UC_2 microspheres at burnups up to 25 at .% heavy metal fissioned^{16,17}. Fast fluence in these experiments was below 10^{20} neutrons/cm² ($E > 0.18$ MEV). The relative degree of shading of the data points represents the approximate percentage of particles with broken coatings found in a given lot on post-irradiation examination. Perfect agreement between theory and experiment would have resulted in only filled (black) points above the diagonal (100% failure) and only open points below (100% survival). Discrepancies result from variations in dimensions and material properties among the batches of coated particles and from inaccurate knowledge of coated-particle properties.

As a second example of comparison between model predictions and experimental results, calculated stress histories are presented for two particles with multi-layer coatings incorporating a silicon carbide diffusion barrier between two structural pyrolytic carbon layers. Calculations were performed using the Walther-Novario program^{10,11}. Figure 12 shows the maximum calculated tangential stress in each structural coating layer during the test particle's fabrication and irradiation history and during a post-irradiation anneal. Note that the calculated stress levels rise smoothly during irradiation until a gap between the inner pyrolytic carbon and silicon carbide layers formed during fabrication closes, transmitting stress from internal fission-gas pressure to the silicon carbide. From this point, the stress rises rapidly until the silicon carbide layer ruptures, then drops to zero. Both pyrolytic carbon layers maintain their integrity until the post-irradiation heating, when the inner layer ruptures at 1500°C. The outer layer remains intact. This particle was irradiated and annealed in an individual compartment to allow its own released fission products, if any, to be detected. Cesium was found outside the particle, indicating that the silicon carbide barrier had, indeed, probably failed. However no krypton was observed, showing that at least one of the pyrolytic-carbon layers had maintained its integrity, as predicted.

Figure 13 shows the stress history of a second coated particle, irradiated in the same experiment. For this particle, neither cesium nor krypton was observed after irradiation, or on post-irradiation heating, indicating that both silicon carbide and pyrolytic-carbon coating layers had remained intact. The stress calculations indicate that this would have been expected, since the stress level in all layers remained below expected rupture values.

DISCUSSION

The apparent ability of these mathematical models to predict failure conditions in reasonable agreement with those observed in an irradiation test is encouraging, but by no means definitive. Accurate independent measurements are still incomplete for several important model parameters, in particular the mechanical properties and failure criteria for the outer coating materials under irradiation, the effective fraction of total porosity made available by the buffer coating materials for fuel expansion and fission-gas accommodation, and the relation of fuel swelling and fission-gas release to temperature and burnup. The interaction between these parameters is complex, and more than one set of reasonable values can provide almost the same predicted fluence at failure.

To place this work on a sounder quantitative footing in order to permit accurate optimization of coated-particle designs, additional data are needed on the mechanical behavior of pyrolytic-carbon coating materials in the range of temperature and fast-neutron flux levels anticipated for high-temperature gas-cooled reactor fuel operation, as well as on the statistical distribution of these properties among commercially-produced lots of coated particles.

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Fig. 1. Model of Fuel Particle with Multilayer Coating of Pyrolytic Carbon and Silicon Carbide.

Fig. 2. Deformation of Spherical Coatings.

Equation of equilibrium for an element of a spherical shell:

$$\frac{d\sigma_r}{dr} + \frac{2}{r} (\sigma_r - \sigma_\theta) = 0 \quad (a < r < b)$$

subject to boundary conditions:

$$\sigma_r(a+) = -P$$

$$\sigma_r(b-) = 0$$

Fig. 3. Equilibrium Equation and Boundary Conditions: σ_r = Radial Stress Component; σ_θ = Tangential Stress Component; P = Fission-Gas Pressure at Inner Surface of Coating ($r = a$).

Compatibility equations are combined with stress-displacement relations for an anisotropic spherical shell whose symmetry axis coincides with the radial direction, to yield:

$$\frac{u}{r} = \left(\frac{1 - \nu_1}{E_1} \right) \sigma_\theta - \left(\frac{\nu_2}{E_2} \right) \sigma_r + g_\theta$$

$$\frac{du}{dr} = \left(\frac{-2\nu_3}{E_1} \right) \sigma_\theta + \left(\frac{1}{E_2} \right) \sigma_r + g_r$$

where, for an elastic body

$$g_\theta = \alpha_\theta (T - T_c) + \eta_\theta$$

$$g_r = \alpha_r (T - T_c) + \eta_r$$

Fig. 4. Displacements in Terms of Stresses: u = Radial Displacement; E_1, E_2 = Young's Moduli in Tangential and Radial Directions; ν_1, ν_2, ν_3 = Poisson's Ratios; α_θ, α_r = Thermal Expansion Coefficients in Tangential and Radial Directions; η_θ, η_r = Unrestrained Dimensional Change from Fast-Neutron Damage; T, T_c = Irradiation and Fabrication Temperatures.

When creep is taken into account, the "g" functions become:

$$g_\theta(r) = \alpha_\theta (T - T_c) + \eta_\theta + \int_0^t \dot{\epsilon}_{\theta c} dt$$

$$g_r(r) = \alpha_r (T - T_c) + \eta_r + \int_0^t \dot{\epsilon}_{rc} dt$$

Note that while T, η_θ , and η_r may not vary significantly with radial position, $\dot{\epsilon}_{\theta c}$ and $\dot{\epsilon}_{rc}$ will generally show strong radial variations.

Fig. 5. Creep Contributions to Displacements: $\dot{\epsilon}_{\theta c}, \dot{\epsilon}_{rc}$ = Tangential and Radial Components of Creep Strain Rate; t = time.

The solution for tangential stress distribution with radially varying g_θ and g_r is:

$$\sigma_\theta \left(\frac{r}{a} \right) = \frac{P}{2} \left[\frac{(n_1 + 2) \left(\frac{b}{a} \right)^{n_2} \left(\frac{r}{a} \right)^{n_1} - (n_2 + 2) \left(\frac{b}{a} \right)^{n_1} \left(\frac{r}{a} \right)^{n_2}}{\left(\frac{b}{a} \right)^{n_1} - \left(\frac{b}{a} \right)^{n_2}} \right] \\ + \frac{E_1}{(1 - \nu_1)(n_1 - n_2)} \left\{ \left[I_1 \left(\frac{b}{a} \right) - I_2 \left(\frac{b}{a} \right) \right] \left[\frac{(n_2 + 2) \left(\frac{r}{a} \right)^{n_2} - (n_1 + 2) \left(\frac{r}{a} \right)^{n_1}}{\left(\frac{b}{a} \right)^{n_1} - \left(\frac{b}{a} \right)^{n_2}} \right] \right. \\ \left. + (n_1 + 2) I_1 \left(\frac{r}{a} \right) - (n_2 + 2) I_2 \left(\frac{r}{a} \right) \right\}$$

Fig. 6. Tangential Stress Distribution in Coating.

The solution for radial stress distribution with radially varying g_θ and g_r is:

$$\sigma_r \left(\frac{r}{a} \right) = -P \left[\frac{\left(\frac{b}{a} \right)^{n_1} \left(\frac{r}{a} \right)^{n_2} - \left(\frac{b}{a} \right)^{n_2} \left(\frac{r}{a} \right)^{n_1}}{\left(\frac{b}{a} \right)^{n_1} - \left(\frac{b}{a} \right)^{n_2}} \right] \\ + \frac{2E_1}{(1 - \nu_1)(n_1 - n_2)} \left\{ \left[I_1 \left(\frac{b}{a} \right) - I_2 \left(\frac{b}{a} \right) \right] \left[\frac{\left(\frac{r}{a} \right)^{n_2} - \left(\frac{r}{a} \right)^{n_1}}{\left(\frac{b}{a} \right)^{n_1} - \left(\frac{b}{a} \right)^{n_2}} \right] \right. \\ \left. + I_1 \left(\frac{r}{a} \right) - I_2 \left(\frac{r}{a} \right) \right\}$$

Fig. 7. Radial Stress Distribution in Coating.

Where n_1 and n_2 are the roots of the quadratic equation

$$n^2 + 3n + 2 \left[1 - \frac{E_1(1 - \nu_2)}{E_2(1 - \nu_1)} \right] = 0$$

and the integral functions, I_1 and I_2 are computed from the relation

$$I_i \left(\frac{r}{a} \right) = \left(\frac{r}{a} \right)^{n_i} g_\theta(1) - g_\theta \left(\frac{r}{a} \right) + \left(\frac{r}{a} \right)^{n_i} \int_1^{\frac{r}{a}} \left[\frac{g_r - (1 + n_i)g_\theta}{y^{n_i+1}} \right] dy; \quad i = 1, 2$$

Fig. 8. Factors in Stress Distribution Equations.

Accurate relations between creep strain rates and stresses will require detailed knowledge of the mechanical behavior of pyrolytic carbon coating materials under irradiation. We have assumed the following simple relations for the spherically symmetric system.

$$\dot{\epsilon}_{\theta c} = \left(\frac{E_1}{E_2} \right) \left(\frac{\dot{\bar{\epsilon}}_c}{\bar{\sigma}_c} \right) \left(\frac{\sigma_\theta - \sigma_r}{2} \right)$$

$$\dot{\epsilon}_{rc} = \left(\frac{E_1}{E_2} \right) \left(\frac{\dot{\bar{\epsilon}}_c}{\bar{\sigma}_c} \right) (\sigma_r - \sigma_\theta)$$

$$\dot{\bar{\epsilon}}_c = \sqrt{\left(\frac{E_2}{E_1} \right) (2\dot{\epsilon}_{\theta c})^2}$$

$$\bar{\sigma}_c = \frac{\dot{\bar{\epsilon}}_c}{c_1 \phi_f}$$

Fig. 9. Assumed Creep Relations: $\dot{\bar{\epsilon}}_c$ = Effective Creep Strain Rate; $\bar{\sigma}_c$ = Effective Creep Stress; c_1 = Steady-State Creep Coefficient; ϕ_f = Fast Neutron Flux.

Fig. 10. Information Flow in Coated-Particle Simulation.

Predicted Burnup Based on 60,000 psi Effective Stress at Failure, 100% Release of Fission Gas from Kernels at High Temperatures, 10% Release at Low Temperatures, 25% of Inner Coating Porosity Available as Free Volume (Except 50% for Porous Inner Coating), Irradiation Creep Rate of 1×10^{-27} in./in.-psi-neutrons/cm² ($E > 0.18$ MeV).

Fig. 11. Comparison of Observed and Predicted Performance of Coated Particles in High-Burnup, Low-Fast-Fluence Irradiation Tests.

Fig. 12. Stress History in Multilayer Coated Particle; Irradiation Followed by Out-of-Pile Heating; Failed Specimen.

Fig. 13. Stress History in Multilayer Coated Particle; Irradiation Followed by Out-of-Pile Heating; Unfailed Specimen.

X A Model for Thermal and Radiation Stress and Strain in Viscoelastic, Orthotropic Cylinders, and Its Application to Borated-Graphite Absorbers*

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An analysis has been conducted to determine stresses and displacements near the center of long viscoelastic cylinders. Stresses arise due to thermal expansion and irradiation-induced dimensional changes which are anisotropic in transverse planes (i.e., planes perpendicular to the axis of geometrical symmetry). The explicit solution for stress is made possible by the assumption of a linear creep law. The logic is shown for a mathematical model that accounts for finite displacements.

The model is used to predict stresses and displacements in borated-graphite absorbers used in the Peach Bottom high-temperature gas-cooled reactor (HTGR). It is predicted that fracture will not occur in the absorbers. This conclusion is presented with the reservation that there is considerable uncertainty regarding irradiation-induced dimensional changes due to the small amount of available data. However, the assumed values for these parameters are believed to be conservative.

INTRODUCTION

Designers of high-temperature gas-cooled reactors (HTGRs) have resorted primarily to borated-graphite materials for thermal-neutron absorbers because of their chemical and dimensional stability in high-temperature, high-fluence environments and their desirable neutronic properties. Typically, the absorber is made from graphite flour and granular B_4C mixed with coal-tar pitch. The body is formed by warm extrusion or warm molding, subjected to carbonizing and graphitizing heat treatments, and machined to final dimensions. The resulting body consists of a graphite matrix containing B_4C inclusions.

Borated-graphite rods formed by extrusion are expected to exhibit improved dimensional stability in the transverse plane when sufficient material is machined from the surfaces. The structure of surface material can be quite anisotropic due to the effects of viscous flow during the forming operation.¹ However, the expense of

machining is high because of tool wear and the loss of waste material, and the amount of machining required to completely remove the anisotropic layers cannot always be performed.

The Peach Bottom HTGR uses absorbers formed by extrusion. The extruded and baked logs were machined to have a 2.31-in. o.d. and a 17-in. length and were bored out to a 1.31-in. i.d. Only small layers were removed from the inner and outer surfaces. Therefore, it was anticipated that some texture would be present in the transverse plane and that fast-neutron irradiation would induce distorting strains in the cylinders.

It is necessary to predict whether fracture or excessive displacements will occur in the absorbers. The purpose of this paper is to develop a mathematical model for the prediction of stress and strain in the absorbers and to present sample calculations for bracketing these parameters.

THE MATHEMATICAL MODEL

The analysis accounts for stresses arising from anisotropic thermal expansion and aniso-

¹J. C. BOKROS, R. J. PRICE, and G. R. TULLY, Jr., *J. Nucl. Mater.*, 15, 33 (1965).

*Reprinted from *Nuclear Science and Engineering*, Vol. 45, pp. 73-85 (1971), by permission of the American Nuclear Society, Inc.

tropic, irradiation-induced dimensional changes and for stress relaxation from irradiation-induced creep. The thick-walled cylinder is assumed to be homogeneous (i.e., the mechanical effects of B_4C inclusions are ignored) and orthotropic (i.e., the property axes of symmetry are orthogonal and, in this case, coincident with the principal axes of the cylinder and the axes of cylindrical coordinates). A uniform temperature distribution is assumed. The ends are free, and it is assumed that the center is sufficiently far away from the ends (~ 3.7 diameters) so that stresses near the center are not influenced by end effects. (This is the condition of generalized plane strain.) It is assumed that Hooke's law, a linear creep law, and the condition of static equilibrium apply. It is also assumed that the elastic properties are isotropic. (This assumption is discussed in the next section.) These conditions and assumptions combined with the condition of strain compatibility will yield total integrodifferential equations between stress and both r (radial position) and γ (fast-neutron fluence) when displacements in the cylinder are small compared with its dimensions. These equations are set up and their solutions derived in Appendix A. The results are as follows (see Appendix A for nomenclature). In the tangential direction, the stress is

$$\sigma_\theta = \frac{1}{2} h_1(x) \left(1 + \ln \frac{r}{a} - \frac{1 + a^2/r^2}{1 - a^2/b^2} \ln \frac{b}{a} \right),$$

in the radial direction,

$$\sigma_r = -\frac{1}{2} h_1(x) \left(\frac{1 - a^2/r^2}{1 - a^2/b^2} \ln \frac{b}{a} - \ln \frac{r}{a} \right),$$

and in the longitudinal direction,

$$\sigma_z = \frac{1}{2} h_2(x) \left[1 + 2 \ln \frac{r}{a} - \frac{2 \ln (b/a)}{1 - a^2/b^2} \right],$$

where

$$\begin{aligned} h_1(x) &= f(x) + \frac{m_1}{2} (1 + \nu) \exp(m_1 x) \\ &\quad \times \int_0^x f(x) \exp(-m_1 x) dx \\ &\quad + \frac{m_2}{2} (1 - \nu) \exp(m_2 x) \\ &\quad \times \int_0^x f(x) \exp(-m_2 x) dx, \\ h_2(x) &= \nu f(x) + \frac{m_1}{2} (1 + \nu) \exp(m_1 x) \\ &\quad \times \int_0^x f(x) \exp(-m_1 x) dx \\ &\quad - \frac{m_2}{2} (1 - \nu) \exp(m_2 x) \\ &\quad \times \int_0^x f(x) \exp(-m_2 x) dx, \end{aligned}$$

$$m_1 = -\frac{1 - \nu_c}{1 - \nu}, \quad m_2 = -\frac{1 + \nu_c}{1 + \nu},$$

$$f(x) = \frac{E}{1 - \nu^2} [(\alpha_r - \alpha_\theta)(T - T_f) + \eta_r - \eta_\theta],$$

and $x = KE\gamma$.

The analysis for stress in terms of r is similar to a large group of analyses of stresses due to the imposition of distorting inelastic strains such as thermal strains (for example, see Chap. 14 of Ref. 2). Similar problems have been analyzed for the case where creep occurs. The solution for stress in terms of time typically involves approximate calculation methods such as finite-element methods,^{3,4} relaxation and effective-modulus methods,⁵ and trial-and-error methods.⁶ However, the assumption of a linear creep law allows the explicit solution to be made. The analysis given in Appendix A is similar to the analysis of a spherical shell given in Ref. 7 except that the cylinder has a lower degree of symmetry, which results in a second-order time equation instead of first-order as for the spherical shell.

A computer code was written for the purpose of performing calculations of stress and strain without the above requirement for small displacements. This was achieved by employing a trial-and-error calculation whereby stresses and total strains are allowed to accumulate in small increments. During each increment in time an iterative scheme is employed in which the geometry of the cylinder is continually updated in accordance with calculated strain until strain converges. The essential features of the code are described in Appendix B.

CALCULATED RESULTS AND DISCUSSION

Calculations of stress and strain were performed for Peach-Bottom-type absorbers. The parameters used for these calculations are given in Table I. The property data represent typical values based on experimental measurements. The value for Poisson's ratio is typical of artificial graphite bodies. The value for Poisson's ratio in

²S. TIMOSHENKO and J. N. GOODIER, *Theory of Elasticity*, McGraw-Hill Book Company, New York (1951).

³T. Y. CHANG and Y. R. RASHID, *Nucl. Eng. Design*, **14**, 181 (1970).

⁴W. H. SUTHERLAND, *Nucl. Eng. Design*, **11**, 269 (1970).

⁵G. L. ENGLAND and M. PHOK, *Nucl. Eng. Design*, **9**, 488 (1969).

⁶J. W. PRADOS and J. L. SCOTT, *Nucl. Appl.*, **3**, 488 (1967).

⁷D. W. STEVENS, *Nucl. Technol.*, **10**, 301 (1971).

TABLE I
Borated-Graphite Dimensions, Typical Properties,
and Operating Conditions

Dimensions and Properties	
Inner radius	0.655 in.
Outer radius	1.155 in.
Length	17 in.
Poisson's ratio	0.2
Poisson's ratio in creep	0.4
Young's modulus	0.7×10^6 psi
Modulus of rupture	3800 psi
Compressive strength	5460 psi
Creep constant	0.9×10^{-27} (psi n/cm ²) ⁻¹
Irradiation-induced fractional-dimensional-change rate	
Tangential direction	-0.00068 (10 ²¹ n/cm ²) ⁻¹
Radial direction	$+0.00288$ (10 ²¹ n/cm ²) ⁻¹
Longitudinal direction	-0.00230 (10 ²¹ n/cm ²) ⁻¹
Coefficient of thermal expansion	
Tangential direction	5.46×10^{-6} (°C) ⁻¹
Radial direction	6.25×10^{-6} (°C) ⁻¹
Longitudinal direction	5.10×10^{-6} (°C) ⁻¹
Operating Conditions	
Temperature of formation	2200°C
Temperature of operation	600°C
Fast-neutron fluence	6×10^{21} n/cm ²

creep is the value suggested for pyrolytic carbon in Ref. 8.

The creep constant was obtained by assuming that the B₄C does not undergo creep and that it acts as a strain diluent (i.e., stress is equal in graphite and B₄C, and therefore the B₄C inclusions do not provide mechanical reinforcement). The Young's modulus predicted from the latter assumption and nominal values for Young's modulus of B₄C and artificial graphite is consistent with the observed Young's modulus (Table I). Since data are lacking on radiation creep in B₄C, it was assumed that B₄C does not creep at 600°C so that the calculations of stress would be conservative. To calculate the creep constant of borated graphite, a B₄C content of 30 vol% and a graphite creep constant of 1.2×10^{-27} (psi n/cm²)⁻¹ (Ref. 9) were used. The units of fluence (n/cm²) are for neutrons with energy >0.18 MeV.

The coefficient of thermal expansion was determined on cylindrical specimens cut with axes parallel to the three primary directions. Expansion

was measured in a helium-purged quartz dilatometer using specimens 0.5 in. in diameter by 0.5 in. long. The coefficient values between 900 and 1000°C were taken because they are expected to be more representative of the average coefficient of expansion during cooling from the high-forming temperatures to the operating temperature than coefficients at lower temperatures.

The crush strength of the thermal expansion specimens was measured subsequent to the expansion measurements by loading the cylinders to failure axially in an Instron Model TTCM-1 testing machine at a cross-head speed of 0.005 in./min. The Young's modulus of the specimens was calculated based on strain inferred from a measurement of cross-head deflection at failure with an appropriate correction for load cell deflection. The results of this work are shown in Table II.

The data from compression tests indicated that the Young's modulus was about 20% lower in the radial direction than in the tangential direction. In an effort to evaluate the effect of this anisotropy, the analysis of Appendix A was repeated, taking into account anisotropy in modulus but neglecting creep. The results are summarized in Appendix C. It was found that lowering the radial modulus by 20% (while holding the tangential modulus constant) caused a 1% increase in maximum stress. (Maximum stress was found to be independent of the longitudinal modulus). Therefore, the added analytical and calculational complexity of including the effects of anisotropy in Young's modulus was not warranted.

Specimens used in flexure tests were cut as cylinders from the walls of an unirradiated neutron absorber. The axes of the specimens were parallel to the extrusion direction. Bending strength measurements were made at room temperature in an Instron Model TTCM-1 testing machine at a cross-head speed of 0.005 in./min. A four-point loading technique with equal spacing of contact points was used to determine flexure strength. The midpoint deflection of the specimen

TABLE II
Results of Compression Tests of Unirradiated
Borated-Graphite Absorber (30 wt% Boron*)

Direction in Extruded Cylinder	Average ^a and Range of Crush Strength (psi)	Young's Modulus ^a (×10 ⁶ psi)
Tangential	5500 ± 190	0.77 ± 0.06
Radial	5440 ± 64	0.60 ± 0.06
Longitudinal	5715 ± 7	0.60 ± 0.11

*Specimen size: 0.5-in. in diameter, 0.5-in. long.

^aTwo determinations.

⁸J. L. KAAE, *J. Nucl. Mater.*, **34**, 206 (1970).

⁹C. R. KENNEDY, "Metals and Ceramics Division Annual Progress Report for Period Ending June 30, 1967," ORNL-4170, Oak Ridge National Laboratory (1967).

was measured with a recording deflectometer during the test.

The modulus of rupture was calculated using the expression

$$MR = \frac{2Pa}{\pi r^3},$$

where

P = total load (lb)

a = distance between contact points [$\frac{1}{3}$ span length (in.)]

r = radius of sample (in.).

Specimen dimensions and results of this work are shown in Table III.

Irradiation-induced dimensional change data were obtained from specimens ($0.2 \times 0.4 \times 0.6$ in.) irradiated in an experimental capsule in the Engineering Test Reactor and from measurements of full-sized control rod absorbers after 300 effective full power days of operation in the Peach Bottom HTGR. Both the experimental specimens and the full-sized absorbers were irradiated in helium at temperatures of 400 to 700°C. Anisotropic irradiation-induced dimensional change perpendicular and parallel to the extrusion direction was detectable in the irradiation experiment specimens. In the full-sized absorbers, the dimensions across the outside diameter were within manufactured tolerance ($\pm 0.4\%$) after irradiation; therefore, changes in diameter were not detectable. However, the length change of the 17-in.-long absorbers was significant and was dependent on accumulated fast neutron exposure. The details of the irradiation work have been reported elsewhere,^{10,11} but for completeness, the data are shown in Fig. 1. The irradiation-induced dimensional change rates in the longitudinal and radial directions shown in Table I represent the slopes of the solid and dashed lines in Fig. 1, respectively. When the lines were drawn in preliminary work, they represented the best fit of available data. Subsequent refined estimates for fast neutron exposure in the Peach Bottom control rod caused the data points from full-size absorbers to be shifted slightly to the position shown. Therefore, the irradiation data showed less anisotropy than that assumed in the model calculations, which tends to make computed stresses and

strains larger than if the more refined data were used.

Dimensional change in the tangential direction could not be measured directly but was obtained by interpolation between the radial and longitudinal values in accordance with observed coefficients of thermal expansion using the method of Simmons¹² and Simmons and Reynolds.¹³ All di-

TABLE III
Results of Flexure Tests of Unirradiated Borated Graphite Absorber Material (30 wt% Boron)

Specimen Total Length of Span (in.) ^a	Average ^b and Range of Modulus of Rupture (psi)
5.7	3845 + 235 - 292
2.6	3820 + 124 - 303

^aDiameter 0.43 in.

^bSix determinations.

Fig. 1. Irradiation-induced dimensional change for Peach Bottom HTGR control rod absorber material in the radial (⊥) and longitudinal (||) directions.

¹⁰O. M. STANSFIELD, "HTGR Base Program Quarterly Progress Report for the Period Ending May 30, 1969," GA-9372, Gulf General Atomic (1969).

¹¹O. M. STANSFIELD, "Examination of Peach Bottom HTGR Control Rod, Control Rod Guide Tube, and Thermally Released Shutdown Rod CO8-01 After 300 Effective Full Power Days of Operation," GA-10196, Gulf General Atomic (1970).

¹²J. H. W. SIMMONS, in *Proceedings of the Third Carbon Conference*, p. 559, Pergamon Press, London and New York (1959).

¹³J. H. W. SIMMONS and W. N. REYNOLDS, "Dimensional Effects in Graphite on Irradiation," *Uranium and Graphite*, Monograph No. 27, p. 75, Institute of Metals (1962).

mensional changes were assumed to vary linearly with fluence because there are insufficient data to infer curvature.

The assumed parameters of Table I were used in the computer code to calculate the radial distribution of stress in the three principal directions. The results are shown in Fig. 2 as a function of fluence. The initial stress distribution is caused by cooling from the forming temperature to the operating temperature. Although the temperature is uniform during operation, distorting strains are induced because of anisotropy in thermal expansion.

Increasing fluence initially causes a reduction in the magnitude of stresses because the distorting strains due to irradiation are in opposition to those due to the above thermal effect. The stresses are everywhere reduced to zero at a fluence of $\sim 0.3 \times 10^{21}$ n/cm². Maximum tensile and compressive stresses occur at the end-of-life at the outer and inner surfaces, respectively, and they are oriented in the tangential direction.

The displacements of the inner and outer surfaces are shown in Fig. 2 and in greater detail in Fig. 3. A shrinkage of slightly over 1% and a small expansion ($\sim 0.1\%$) are calculated for the

inner and outer surfaces, respectively. The displacements were found to be independent of Young's modulus and the creep constant. However, they are directly proportional to the radial and tangential values of irradiation-induced dimensional change when displacement is small. Displacement is linearly dependent on fluence because irradiation-induced dimensional change was assumed to vary linearly with fluence.

The influence of the creep constant and Young's modulus on maximum stress was investigated by means of a parametric study. A series of calculations was performed for various combinations of these parameters. The results are plotted in two ways in Fig. 4.

When the creep constant is zero, stress is directly proportional to Young's modulus. Near the expected values of Young's modulus and creep constant [0.7×10^6 psi and 0.9×10^{-27} (psi n/cm²)⁻¹, respectively], stress is insensitive to changes in Young's modulus and moderately sensitive to changes in the creep constant. In addition, the stresses in this region are well within the modulus-of-rupture and compressive-strength limits given in Table I. Therefore, fracture is not expected; however, these results must be inter-

Fig. 2a. The fluence history of the radial distribution of stress in the tangential direction.

Fig. 2b. The fluence history of the radial distribution of stress in the radial direction.

Fig. 2c. The fluence history of the radial distribution of stress in the longitudinal direction.

preted with caution, mainly because of uncertainty in irradiation-induced dimensional change. While it is believed that the assumed values for this parameter are conservative, they are based on

very few data. In addition, the effects of the $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction were assumed to be insignificant. This reaction apparently caused accelerated dimensional change and bowing due to ^{10}B burnup

Fig. 3. Fractional displacement of inner and outer surfaces vs fluence.

gradients in borated graphite made with ^{10}B enriched boron,¹⁴ but in the same experiments, specimens of the Peach Bottom absorber material showed no effects of the ^{10}B burnup gradient.

SUMMARY

An explicit solution was obtained for stresses and displacements in the central portion of long thick-walled cylinders resulting from the distorting strains set up by anisotropic thermal-expansion and irradiation-induced dimensional change. The analysis accounts for stress relaxation by irradiation-induced creep, and the solution was put into the form of a computer code that accounts for finite displacements.

It was predicted that fracture in Peach Bottom absorbers would not occur. However, this conclusion must be interpreted with caution because

¹⁴O. M. STANSFIELD, "HTGR Base Program Quarterly Progress Report for the Period Ending November 30, 1970," GA-10399, Gulf General Atomic (1970).

A. TANGENTIAL STRESS VS. YOUNG'S MODULUS

B. TANGENTIAL STRESS VS. CREEP CONSTANT

Fig. 4. The influence of Young's modulus and creep constant on maximum and minimum tangential stress. Stress is plotted vs (a) Young's modulus, and (b) creep constant for a fluence of 6×10^{21} n/cm².

of uncertainties due to the small amount of data for irradiation-induced dimensional change in borated graphite materials.

APPENDIX A

DERIVATION OF THERMAL AND RADIATION STRESS AND STRAIN IN VISCOELASTIC ORTHOTROPIC CYLINDERS

Summary

The derivation of stress and strain near the center of a long thick-walled orthotropic cylinder is presented. Stresses arising from anisotropic thermal expansion and anisotropic irradiation-induced dimensional changes are considered; however, the effects of applying a pressure can be added by superposition. The analysis also considers stress relaxation by irradiation-induced creep.

The following conditions are assumed to apply: Hooke's law, a linear creep law, static equilibrium, and generalized plane strain in the longitudinal direction. These conditions along with the condition of strain compatibility yield total integrodifferential equations between stress and both radial position and pseudo-time (fast-neutron fluence) when infinitesimal displacements are assumed. These relationships are solved in this appendix,¹⁵ and the assumption of infinitesimal displacements is removed by means of an iterative computation scheme described in Appendix B.

THE DEVELOPMENT OF THE INTEGRO-DIFFERENTIAL STRESS EQUATION

The assumed interrelationships of stresses and strains are

Hooke's Law

$$e_{\theta} = \frac{\sigma_{\theta}}{E} - \frac{\nu}{E} (\sigma_r + \sigma_z)$$

$$e_r = \frac{\sigma_r}{E} - \frac{\nu}{E} (\sigma_{\theta} + \sigma_z)$$

$$e_z = \frac{\sigma_z}{E} - \frac{\nu}{E} (\sigma_{\theta} + \sigma_r)$$

Creep Law

$$\dot{e}_{c\theta} = K\sigma_{\theta} - K\nu_c (\sigma_r + \sigma_z)$$

$$\dot{e}_{cr} = K\sigma_r - K\nu_c (\sigma_{\theta} + \sigma_z)$$

$$\dot{e}_{cz} = K\sigma_z - K\nu_c (\sigma_{\theta} + \sigma_r)$$

¹⁵Nomenclature is given at the end of this Appendix (A).

Total Strain

$$\epsilon_{\theta} = e_{\theta} + \alpha_{\theta} (T - T_f) + \eta_{\theta} + \int_0^{\gamma} \dot{e}_{c\theta} d\gamma$$

$$\epsilon_r = e_r + \alpha_r (T - T_f) + \eta_r + \int_0^{\gamma} \dot{e}_{cr} d\gamma$$

$$\epsilon_z = e_z + \alpha_z (T - T_f) + \eta_z + \int_0^{\gamma} \dot{e}_{cz} d\gamma$$

Equilibrium

$$\sigma_r' + \frac{\sigma_r - \sigma_{\theta}}{r} = 0$$

$$\int_a^b r \sigma_z dr = 0$$

Compatibility

$$\epsilon_{\theta} = \frac{u}{r}$$

$$\epsilon_r = \frac{du}{dr}$$

Condition of Generalized Plane Strain

$$\epsilon_z = k(\gamma)$$

In the expressions for total strain, the variable u can be eliminated by noting that $d(r\epsilon_{\theta})/dr = du/dr$, yielding

$$\begin{aligned} 0 &= \frac{r\sigma_{\theta}'}{E} - \frac{\nu r}{E} (\sigma_r' + \sigma_z') + Kr \\ &\times \int_0^{\gamma} [\sigma_{\theta}' - \nu_c (\sigma_r' + \sigma_z')] d\gamma + \frac{1+\nu}{E} (\sigma_{\theta} - \sigma_r) \\ &+ (\alpha_{\theta} - \alpha_r)(T - T_f) + \eta_{\theta} - \eta_r + K(1 + \nu_c) \\ &\times \int_0^{\gamma} (\sigma_{\theta} - \sigma_r) d\gamma \end{aligned}$$

Since $\sigma_{\theta} - \sigma_r = r\sigma_r'$,

$$\begin{aligned} \frac{r}{E} (\sigma_{\theta}' + \sigma_r' - \nu\sigma_z') + Kr \int_0^{\gamma} (\sigma_{\theta}' + \sigma_r' - \nu_c \sigma_z') d\gamma \\ = (\alpha_r - \alpha_{\theta})(T - T_f) + \eta_r - \eta_{\theta} \end{aligned} \quad (1)$$

Differentiating the expression for ϵ_z and solving the integral equation in dose will allow the elimination of σ_z' ,

$$\begin{aligned} \epsilon_z' = 0 &= \frac{\sigma_z'}{E} - \frac{\nu}{E} (\sigma_{\theta}' + \sigma_r') + K \\ &\times \int_0^{\gamma} [\sigma_z' - \nu_c (\sigma_{\theta}' + \sigma_r')] d\gamma \end{aligned} \quad (2)$$

The solution for σ_z' is obtained by differentiating with respect to γ and solving the differential equation in dose, yielding

$$\begin{aligned} \sigma_z' &= \nu (\sigma_{\theta}' + \sigma_r') + KE(\nu_c - \nu) \exp(-KE\gamma) \\ &\times \int_0^{\gamma} (\sigma_{\theta}' + \sigma_r') \exp(KE\gamma) d\gamma \end{aligned} \quad (3)$$

Inserting Eq. (3) into Eq. (2) yields

$$\int_0^Y \sigma'_z d\gamma = \nu_c \int_0^Y (\sigma'_\theta + \sigma'_r) d\gamma - (\nu_c - \nu) \exp(-KE\gamma) \times \int_0^Y (\sigma'_\theta + \sigma'_r) \exp(KE\gamma) d\gamma \quad (4)$$

Inserting Eqs. (3) and (4) into Eq. (1) yields

$$\begin{aligned} \frac{1-\nu^2}{E} r(\sigma'_\theta + \sigma'_r) + K(1-\nu^2) r \int_0^Y (\sigma'_\theta + \sigma'_r) d\gamma \\ + K(\nu_c - \nu)^2 r \exp(-KE\gamma) \\ \times \int_0^Y (\sigma'_\theta + \sigma'_r) \exp(KE\gamma) d\gamma \\ = (\alpha_r - \alpha_\theta)(T - T_f) + \eta_r - \eta_\theta \quad (5) \end{aligned}$$

The expression $[r(\sigma'_\theta + \sigma'_r)]$ becomes $(r^2\sigma''_r + 3r\sigma'_r)$, since by differentiation of the σ_r, σ_θ equilibrium condition, $\sigma'_\theta = r\sigma''_r + 2\sigma'_r$. By setting $Y = r^2\sigma''_r + 3r\sigma'_r$, Eq. (5) becomes

$$Y + \frac{1-\nu^2}{1-\nu^2} \int_0^Y Y dx + \frac{(\nu_c - \nu)^2}{1-\nu^2} e^{-x} \int_0^x Y e^x dx = f(x), \quad (6)$$

where

$$f(x) = \frac{E}{1-\nu^2} [(\alpha_r - \alpha_\theta)(T - T_f) + \eta_r - \eta_\theta],$$

and the fluence variable has been transformed to $x = KE\gamma$.

SOLUTION OF THE STRESS EQUATION IN FLUENCE

Equation (6) can be converted to a second-order differential equation in x by successive differentiation; however, its form suggests the use of Laplace transforms to obtain its solution. Accordingly, the transform of Eq. (6) is

$$\begin{aligned} \Lambda(Y) + \frac{1-\nu^2}{1-\nu^2} \frac{\Lambda(Y)}{S} + \frac{(\nu_c - \nu)^2}{1-\nu^2} \\ \times \left[\Lambda \left(\int_0^x Y e^x dx \right) \right]_{S \rightarrow S+1} = \Lambda(f), \\ \left[1 + \frac{1-\nu^2}{1-\nu^2} \frac{1}{S} + \frac{(\nu_c - \nu)^2}{1-\nu^2} \frac{1}{S+1} \right] \Lambda(Y) = \Lambda(f), \end{aligned}$$

which reduces to

$$\Lambda(Y) = \Lambda(f) \frac{S^2 + S}{(S - m_1)(S - m_2)}, \quad (7)$$

where

$$m_1 = -\frac{1-\nu_c}{1-\nu}, \quad m_2 = -\frac{1+\nu_c}{1+\nu}.$$

Now since

$$\frac{1}{(S - m_1)(S - m_2)} = \Lambda[\exp(m_1 x)] \Lambda[\exp(m_2 x)],$$

the application of the convolution theorem yields

$$\begin{aligned} \Lambda[\exp(m_1 x)] \Lambda[\exp(m_2 x)] \\ = \Lambda \left\{ \int_0^x \exp[m_1(x - \lambda)] \exp(m_2 \lambda) d\lambda \right\} \\ = \Lambda \left[\frac{\exp(m_2 x) - \exp(m_1 x)}{m_2 - m_1} \right], \end{aligned}$$

where λ is a dummy variable. Then Eq. (7) becomes

$$\Lambda(Y) = \Lambda(f)(S^2 + S) \Lambda \left[\frac{\exp(m_1 x) - \exp(m_2 x)}{m_1 - m_2} \right]. \quad (8)$$

Denoting

$$\begin{aligned} g(x) &= \frac{\exp(m_1 x) - \exp(m_2 x)}{m_1 - m_2}, \\ \dot{g} &= \frac{dg}{dx} = \frac{m_1 \exp(m_1 x) - m_2 \exp(m_2 x)}{m_1 - m_2}, \\ \ddot{g} &= \frac{m_1^2 \exp(m_1 x) - m_2^2 \exp(m_2 x)}{m_1 - m_2}. \end{aligned}$$

Observing that $g(0) = 0$ and $\dot{g}(0) = 1$, one finds

$$\begin{aligned} S\Lambda(g) &= \Lambda \left[\frac{m_1 \exp(m_1 x) - m_2 \exp(m_2 x)}{m_1 - m_2} \right] + g(0) \\ &= \Lambda \left[\frac{m_1 \exp(m_1 x) - m_2 \exp(m_2 x)}{m_1 - m_2} \right], \end{aligned}$$

and

$$\begin{aligned} S^2\Lambda(g) &= \Lambda \left[\frac{m_1^2 \exp(m_1 x) - m_2^2 \exp(m_2 x)}{m_1 - m_2} \right] \\ &\quad + Sg(0) + \dot{g}(0) \\ &= \Lambda \left[\frac{m_1^2 \exp(m_1 x) - m_2^2 \exp(m_2 x)}{m_1 - m_2} \right] + 1. \end{aligned}$$

Equation (8) now becomes

$$\begin{aligned} \Lambda(Y) &= \Lambda(f) \left\{ \Lambda \left[\frac{m_1 \exp(m_1 x) - m_2 \exp(m_2 x)}{m_1 - m_2} \right] \right. \\ &\quad \left. + \Lambda \left[\frac{m_1^2 \exp(m_1 x) - m_2^2 \exp(m_2 x)}{m_1 - m_2} \right] + 1 \right\} \\ &= \Lambda(f) \left\{ \Lambda \left[m_1 \frac{1+m_1}{m_1 - m_2} \exp(m_1 x) \right. \right. \\ &\quad \left. \left. - m_2 \frac{1+m_2}{m_1 - m_2} \exp(m_2 x) \right] + 1 \right\}. \end{aligned}$$

Noting that

$$\begin{aligned} \frac{1+m_1}{m_1 - m_2} &= \frac{1}{2} (1 + \nu) \quad \text{and} \quad \frac{1+m_2}{m_1 - m_2} \\ &= -\frac{1}{2} (1 - \nu), \end{aligned}$$

and applying the convolution theorem, one obtains

$$\begin{aligned}\Lambda(Y) &= \Lambda(f) + \Lambda\left\{\frac{m_1}{2}(1+\nu)\right. \\ &\quad \times \int_0^x f(\lambda) \exp[m_1(x-\lambda)]d\lambda + \frac{m_2}{2}(1-\nu) \\ &\quad \times \left.\int_0^x f(\lambda) \exp[m_2(x-\lambda)]d\lambda\right\} \\ &= \Lambda(f) + \Lambda\left[\frac{m_1}{2}(1+\nu)\exp(m_1x)\right. \\ &\quad \times \int_0^x f \exp(-m_1x)dx + \frac{m_2}{2}(1-\nu)\exp(m_2x) \\ &\quad \times \left.\int_0^x f \exp(-m_2x)dx\right].\end{aligned}$$

The inverse transform can now be made, which yields

$$\begin{aligned}Y &= f + \frac{m_1}{2}(1+\nu)\exp(m_1x) \\ &\quad \times \int_0^x f \exp(-m_1x)dx + \frac{m_2}{2}(1-\nu)\exp(m_2x) \\ &\quad \times \int_0^x f \exp(-m_2x)dx.\end{aligned}\quad (9)$$

Some special cases which may be of interest are

When $\nu_c = \nu$, $m_1 = m_2 = -1$, and

$$Y = f - e^{-x} \int_0^x f e^x dx;$$

When $f = f_0 = \text{constant}$,

$$Y = \frac{f_0}{2} \left[(1+\nu)\exp(m_1x) + (1-\nu)\exp(m_2x) \right];$$

When $\nu_c = \nu$ and $f = f_0$,

$$Y = f_0 e^{-x}.$$

Equation (9) can also be expressed in the equivalent form

$$\begin{aligned}Y &= f(0) \left[\frac{m_1}{m_1 - m_2} \exp(m_1x) - \frac{m_2}{m_1 - m_2} \exp(m_2x) \right] \\ &\quad + \int_0^x [f(\lambda) + f(\lambda)] \\ &\quad \times \frac{m_1 \exp[m_1(x-\lambda)] - m_2 \exp[m_2(x-\lambda)]}{m_2 - m_1} d\lambda.\end{aligned}$$

THE SOLUTION OF THE STRESS EQUATION IN r

To return to the differential equation in r , define the right side of Eq. (9) as $h_1(x)$,

$$Y = r^2 \sigma_r'' + 3r \sigma_r' = h_1(x).$$

This Euler equation has the solution

$$\sigma_r = -\frac{1}{2} h_1(x) \left(\frac{1 - a^2/r^2}{1 - a^2/b^2} \ln \frac{b}{a} - \ln \frac{r}{a} \right), \quad (10)$$

where the following boundary conditions have been applied:

$$\sigma_r = 0 \text{ at } r = a \text{ (inner radius)},$$

$$\sigma_r = 0 \text{ at } r = b \text{ (outer radius)}.$$

Inserting Eq. (10) (and its derivative) into the σ_r, σ_θ condition of equilibrium yields

$$\sigma_\theta = \frac{1}{2} h_1(x) \left(1 + \ln \frac{r}{a} - \frac{1 + a^2/r^2}{1 - a^2/b^2} \ln \frac{b}{a} \right). \quad (11)$$

The position of the critical value of σ_r is $r_{cr} = \left(\ln \frac{b}{a} \frac{2a^2b^2}{b^2 - a^2} \right)^{1/2}$, and that of σ_θ has the same magnitude but is imaginary. Therefore, σ_θ varies monotonically with r , and its most severe values are at $r = a$ and/or $r = b$.

At $r = a$,

$$\sigma_\theta(a) = h_1(x) \left(\frac{1}{2} - \frac{\ln \frac{b}{a}}{1 - a^2/b^2} \right).$$

At $r = b$,

$$\sigma_\theta(b) = h_1(x) \left(\frac{1}{2} - \frac{a^2/b^2}{1 - a^2/b^2} \ln \frac{b}{a} \right).$$

THE SOLUTION FOR σ_z

The solution of σ_z follows in a manner analogous to the above derivations.

By adding Eqs. (10) and (11),

$$\sigma_\theta + \sigma_r = \frac{1}{2} h_1(x) \left[1 + 2 \ln \left(\frac{r}{a} \right) - \ln \left(\frac{b}{a} \right) \frac{2b^2}{b^2 - a^2} \right],$$

and

$$r(\sigma_\theta' + \sigma_r') = h_1(x).$$

Inserting this into Eq. (3)

$$r \sigma_z' = \nu h_1 + (\nu_c - \nu) e^{-x} \int_0^x h_1 e^x dx. \quad (12)$$

When the expression for h_1 [Eq. (9)] is inserted, this equation appears to be quite complicated. However, it can be simplified by the use of Laplace transforms. Defining the right side of Eq. (12) as $h_2(x)$, the Laplace of h_2 is obtained in a manner similar to the development of Eq. (8)

$$\Lambda(h_2) = \Lambda(h_1) \frac{\nu S + \nu_c}{S + 1}.$$

Inserting Eq. (8) into this expression

$$\Lambda(h_2) = \Lambda(f) (\nu S^2 + \nu_c S) \Lambda \left[\frac{\exp(m_1x) - \exp(m_2x)}{m_1 - m_2} \right].$$

The solution for h_2 now becomes analogous to the derivation of h_1 from Eqs. (8) through (9). The

result is

$$h_2(x) = \nu f + \frac{m_1}{2} (1 + \nu) \exp(m_1 x) \\ \times \int_0^x f \exp(-m_1 x) dx - \frac{m_2}{2} (1 - \nu) \\ \times \exp(m_2 x) \int_0^x f \exp(-m_2 x) dx \quad (13)$$

Some special cases are:

When $\nu_c = \nu$, $m_1 = m_2 = -1$, and

$$h_2 = \nu f - \nu e^{-x} \int_0^x f e^x dx \\ = \nu h_1 ;$$

When $f = f_0 = \text{constant}$,

$$h_2 = \frac{f_0}{2} [(1 + \nu) \exp(m_1 x) - (1 - \nu) \exp(m_2 x)] ;$$

When $\nu_c = \nu$ and $f = f_0$,

$$h_2 = \nu f_0 e^{-x} .$$

Equation (13) can also be expressed in the equivalent form

$$h_2 = \nu f(0) \left[\frac{m_1}{m_1 - m_2} \exp(m_1 x) - \frac{m_2}{m_1 - m_2} \exp(m_2 x) \right] \\ + \int_0^x [\nu \dot{f}(\lambda) + \nu_c f(\lambda)] \\ \times \frac{m_1 \exp[m_1(x - \lambda)] - m_2 \exp[m_2(x - \lambda)]}{m_1 - m_2} d\lambda .$$

The solution of the differential equation in r [Eq. (12)] is readily obtained by integration,

$$\sigma_z - \sigma_z(a) = h_2(x) \ln \left(\frac{r}{a} \right) .$$

Now applying the σ_z condition of equilibrium gives

$$\sigma_z(a) = h_2(x) \left(\frac{1}{2} - \frac{\ln \frac{b}{a}}{1 - a^2/b^2} \right) ,$$

and the solution is

$$\sigma_z = \frac{1}{2} h_2(x) \left(1 + 2 \ln \frac{r}{a} - \frac{2 \ln \frac{b}{a}}{1 - a^2/b^2} \right) . \quad (14)$$

$$\text{At } r = b, \sigma_z(b) = h_2(x) \left(\frac{1}{2} - \frac{a^2/b^2}{1 - a^2/b^2} \ln \frac{b}{a} \right) .$$

Note that σ_z increases or decreases monotonically with r depending on the sign of h_2 .

The effect of a uniform internal pressure can be superimposed if desired by adding the corresponding Lamé equations for thick-walled cylinders to Eqs. (10), (11), and (14).

Total strains can now be obtained from Hooke's law and the creep law. At the inner surface,

$$\epsilon_\theta(a) = \left[\frac{1}{2} - \frac{\ln(b/a)}{1 - a^2/b^2} \right] [(\alpha_r - \alpha_\theta)(T - T_f) \\ + \eta_r - \eta_\theta] + \alpha_\theta(T - T_f) + \eta_\theta ;$$

and at the outer surface,

$$\epsilon_\theta(b) = \left[\frac{1}{2} - \frac{a^2/b^2}{1 - a^2/b^2} \ln \frac{b}{a} \right] [(\alpha_r - \alpha_\theta)(T - T_f) \\ + \eta_r - \eta_\theta] + \alpha_\theta(T - T_f) + \eta_\theta .$$

Note that total strain is independent of all elastic and creep parameters. However, this will not be true if an internal pressure is imposed.

NOMENCLATURE

a	= inner radius
b	= outer radius
e	= elastic strain
E	= Young's modulus
K	= creep constant
r, θ, z	= cylindrical coordinates
S	= the independent variable in the Laplace transform of a function
T	= operating temperature
T_f	= temperature at which cylinder is formed (i.e., is stress-free)
u	= radial displacement
α	= coefficient of linear thermal expansion
γ	= fast-neutron fluence (energy > 0.18 MeV)
ϵ	= total strain
ϵ_c	= creep strain
η	= fractional irradiation-induced dimensional change
Λ	= Laplace transform operator
ν	= Poisson's ratio
ν_c	= "Poisson's ratio in creep"
σ	= stress

APPENDIX B

DESCRIPTION OF COMPUTER MODEL

The solutions for stress given in Appendix A are explicit, and the aid of a computer is not necessary as long as the assumption of infinitesimal displacements is valid and an analytical expression for $f(x)$ is available such that the integrals of $f \exp(-m_1 x)$ and $f \exp(-m_2 x)$ can be

evaluated analytically. A computer code was written for the purpose of removing the assumption of infinitesimal displacements. A flow chart for this code is given in Fig. 5. In essence, the code functions as follows:

1. After incrementing fluence, stresses are calculated using the solutions given in Appendix A.

2. The fluence increment is sufficiently small so that numeric integration of stresses over fluence are obtained to the desired degree of accuracy. These integrals are used in the creep law to obtain creep strain in the tangential direction. Total tangential strain (ϵ_θ) can now be calculated from Hooke's law and known values of creep, thermal, and irradiation-induced strains.

3. The original radial dimensions of the cylinder are now increased by the factors $[1 + \epsilon_\theta(a)]$ and $[1 + \epsilon_\theta(b)]$ at the inner and outer surfaces, respectively. Stresses are recalculated using the corrected radial dimensions.

4. Tangential strain is recalculated, and if it differs from the previous value by less than some small, pre-assigned value, convergence is obtained and we return to step 1. If convergence is not obtained, a modified Newton method¹⁶ is used to make a guess for tangential strain and we return to step 3. This iterative method uses the

¹⁶ J. H. WEGSTEIN, "Accelerating Convergence of Iterative Processes," *Commun. Assoc. Computing Machinery*, 1, 9 (1958).

Fig. 5. Flow chart for computer code for stress and strain in a cylinder.

observed relationship between previous guesses for strain and the resulting calculations of strain to speed convergence.

APPENDIX C

EQUATIONS FOR THERMAL AND RADIATION STRESS AND STRAIN IN ORTHOTROPIC CYLINDERS WITH ANISOTROPIC YOUNG'S MODULUS

The stress analysis of Appendix A is repeated in this section taking into account anisotropy in Young's modulus but assuming that creep does not occur. Only a summary is given because the analysis is straightforward.

The starting equations (and nomenclature) are the same as in Appendix A, except that the creep constant is set equal to zero and Hooke's law is rewritten to account for anisotropy in Young's modulus as follows:

$$e_{\theta} = \frac{\sigma_{\theta}}{E_{\theta}} - \nu \left(\frac{\sigma_r}{E_r} + \frac{\sigma_z}{E_z} \right),$$

$$e_r = \frac{\sigma_r}{E_r} - \nu \left(\frac{\sigma_{\theta}}{E_{\theta}} + \frac{\sigma_z}{E_z} \right),$$

$$e_z = \frac{\sigma_z}{E_z} - \nu \left(\frac{\sigma_{\theta}}{E_{\theta}} + \frac{\sigma_r}{E_r} \right).$$

The resulting spatial differential equation is

$$r^2 \sigma_r'' + (3 + \nu \delta) r \sigma_r' + \delta \sigma_r = f(x),$$

where

$$\delta = \frac{E_{\theta}}{1 - \nu} \left(\frac{1}{E_{\theta}} - \frac{1}{E_r} \right)$$

and $f(x)$ has the same meaning as before, except E is replaced by E_{θ} . The stress solutions for $E_{\theta} \neq E_r$ are

$$\sigma_r = \frac{f}{\mu_1} \left[\frac{\frac{a^{\mu_2} - b^{\mu_2}}{\mu_2} (a^{\mu_1} - r^{\mu_1}) + \frac{r^{\mu_2} - a^{\mu_2}}{\mu_2} (a^{\mu_1} - b^{\mu_1})}{a^{\mu_2} b^{\mu_1} - a^{\mu_1} b^{\mu_2}} \right],$$

$$\sigma_{\theta} = \frac{f}{\mu_1} \left\{ \frac{\frac{a^{\mu_2} - b^{\mu_2}}{\mu_2} [a^{\mu_1} - (1 - \mu_1) r^{\mu_1}] + \frac{r^{\mu_2} - a^{\mu_2}}{\mu_2} (a^{\mu_1} - b^{\mu_1}) + r^{\mu_2} (a^{\mu_1} - b^{\mu_1})}{a^{\mu_2} b^{\mu_1} - a^{\mu_1} b^{\mu_2}} \right\},$$

where

$$\mu_1 = -\frac{2 + \nu \delta}{2} \left[1 + \left(1 - \frac{4\delta^2}{(2 + \nu \delta)^2} \right)^{1/2} \right],$$

and

$$\mu_2 = -\frac{2 + \nu \delta}{2} \left[1 - \left(1 - \frac{4\delta^2}{(2 + \nu \delta)^2} \right)^{1/2} \right].$$

The solutions of Appendix A (with zero creep) can be obtained from the above solutions by noting that as $E_{\theta} \rightarrow E_r$, $\mu_1 \rightarrow -2$ and $\mu_2 \rightarrow 0$ and then applying

Fig. 6. Influence of radial Young's modulus on tangential stress at the outer surface using $E_{\theta} = 0.7 \times 10^6$ psi. Tangential stress is given as the ratio to the value for the isotropic case.

the relationship

$$\lim_{\mu_2 \rightarrow 0} \frac{a^{\mu_2} - b^{\mu_2}}{\mu_2} = \ln \frac{a}{b} \quad \text{for } ab > 0.$$

Values for σ_{θ} (which are the highest tensile stresses) were calculated using the input data of Table I and using $E_{\theta} = 0.7 \times 10^6$ psi. The results are compared to the case $E_{\theta} = E_r$ in Fig. 6 with E_r as the independent variable. It is seen that σ_{θ}

is very insensitive to E_r (although σ_{θ} is nearly proportional to E_{θ}). For $E_r = 0.56 \times 10^6$ psi (which is 20% below E_{θ}), σ_{θ} is <1% higher than for $E_r = 0.7 \times 10^6$ psi.

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STRUCTURAL DESIGN OF NUCLEAR ROCKET FUEL ELEMENTS

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ABSTRACT

This paper presents a status report on NERVA fuel element analytic techniques, including stress calculations in a complicated stress field by finite element methods, the problem of defining a satisfactory criterion of failure, and the application of state-of-the-art probabilistic techniques in predicting the fuel reliability.

The energy source in the NERVA nuclear rocket engine is a high power density reactor that operates at an impressive temperature level (nozzle chamber temperature of 4250°R) and uses hydrogen as the propellant. The fuel elements are graphite, fueled by enriched uranium carbide which is dispersed through the volume in bead form or in solid solution with another carbide. The elements are hexagonal, about three-quarters of an inch across flats, about 52 inches long, and contain 19 circular coolant channels. At the NERVA operating temperatures, the carbon in the fuel elements is highly reactive with the hydrogen propellant, with hydrocarbons like methane and acetylene formed and exhausted at the expense of fueled material. As a corrosion reduction measure, the fuel is coated with a refractory carbide.

The principal causes for stresses in the elements are: (1) the high power density, (2) the interaction between the fuel matrix and the corrosion resistant carbide coating, and (3) the interaction between adjacent elements due to inevitable element-to-element temperature differences. The method of calculating the combined stress field due to these mechanisms and others of less importance is discussed.

NOTE: The Nuclear Engine for Rocket Vehicle Application Program (NERVA) is administered by the Space Nuclear Propulsion Office (SNPO), a joint office of the U. S. Atomic Energy Commission and the National Aeronautics and Space Administration. Aerojet Nuclear Systems Company as prime contractor for the engine system and Westinghouse Electric Corporation as subcontractor for the nuclear subsystem, are developing a nuclear propulsion system for space applications.

The conversion of the calculated stresses to a safety factor or a reliability by means of an adequate criterion of failure is discussed. The possible criteria of failure for the brittle graphite elements are explored with comments on the feasibility of each criterion. Possible approaches are: (1) fracture mechanics, (2) Weibull (weakest link) statistics, (3) maximum principal stress, (4) maximum principal strains, and (5) a strain energy criterion.

The final topic is a description of the probabilistic techniques incorporated into the NERVA fuel stress analysis. Both the mean values and variances of the individual stress components are calculated and are then combined statistically to define the failure-governing stress. This stress and the component strength, both defined probabilistically, are subsequently compared by the "interference theory" or "stress-strength overlap" method to predict the fuel reliability.

I. INTRODUCTION

This paper presents a status report on NERVA fuel element analytic techniques, including stress calculations in a complicated stress field by finite element methods, the problem of defining a satisfactory criterion of failure, and the application of state-of-the-art probabilistic techniques in predicting the reliability of the fuel.

The energy source of the NERVA nuclear rocket engine is a high-power-density reactor that operates at an impressive temperature level (nozzle chamber temperature of 4250°R) and uses hydrogen as the propellant. The NERVA nuclear subsystem is shown in cutaway form in Figure 1. chamber temperature of 4250°R) and uses hydrogen as the propellant. The NERVA nuclear subsystem is shown in cutaway form in Figure 1.

The fuel configuration is shown in Figure 2. The fuel elements are graphite, fueled by enriched uranium carbide which is dispersed through the element volume in bead form or in solid solution with another carbide. The elements are hexagonal, about three-quarters of an inch across flats, about 52 inches long, and contain 19 circular coolant channels. At the NERVA operating temperatures, the carbon in the fuel elements is highly reactive with the hydrogen propellant, with hydrocarbons like methane and acetylene formed and exhausted at the expense of fueled material. As a corrosion reduction measure, the fuel is coated with a refractory carbide. The presence of the carbide coating, as a material differing in stiffness and thermal expansion from the fuel but intimately attached to the fuel, naturally complicates the fuel stress analysis.

The principal causes for stresses in the elements are: (1) the high power density, (2) the interaction between the fuel matrix and the corrosion-resistant carbide coating, and (3) the interaction between adjacent elements due to inevitable element-to-element temperature differences.

FUEL STRESS MECHANISMS

First discussing the stress mechanisms contributing to transverse stress in the element, there is the internal heat generation stress, represented by the expression

$$\sigma_{q'''} = \frac{E\alpha}{k} \cdot q''' \cdot \psi ,$$

where

- ψ = $F(L^2)$, geometric shape factor,
 σ_q''' = maximum thermal stress,
 E = modulus of elasticity,
 α = coefficient of thermal expansion,
 k = thermal conductivity,
 q''' = heat generation rate,
 L = characteristic length.

The other large contributor to element transverse stresses is the coating-matrix interaction stress. The expression for this stress component is

$$\sigma_{c \cdot m} = E_m \left[-\alpha_m \Delta T + \Delta\alpha T_c - \Delta\alpha_d T_d \right] \cdot \phi,$$

where

- $\sigma_{c \cdot m}$ = coating-matrix interaction stress in the matrix,
 E_m = matrix modulus of elasticity at the matrix average temperature, T_m ,
 α_m = matrix coefficient of thermal expansion at temperature T_m ,
 ΔT = temperature difference between the matrix and the coating = $T_m - T_c$,
 T_m = average matrix temperature,
 T_c = average coating temperature,
 $\Delta\alpha$ = difference in coefficient of thermal expansion between matrix and coating = $\alpha_c - \alpha_m$,
 α_c = coating coefficient of thermal expansion at temperature T_c ,
 $\Delta\alpha_d$ = difference in coefficient of thermal expansion between matrix and coating at coating deposition temperature $T_d = \alpha_{cd} - \alpha_{md}$,
 T_d = coating deposition temperature,
 ϕ = geometry and relative stiffness factor between coating and matrix.

Our present best model of this complicated stress component is based on three assumptions:

- a. The coating and substrate interact elastically with one another.
- b. The coating is perfectly adherent.
- c. The coating-matrix assembly is in a stress-free condition at the temperatures which prevailed during the final coating deposition process (a chemical vapor deposition operation).

Since, as has been mentioned, the fuel in the graphite elements is in the form of beads, there is a fuel bead-matrix interaction stress, calculated by the expression

$$\sigma_{fb} = E_m \left[\Delta\alpha (T_m - T_g) + \alpha_b \cdot \Delta T \right] \cdot \theta \text{ for } T_m \geq T_g,$$

where

- σ_{fb} = fuel bead-matrix interaction stress in the matrix,
- E_m = matrix modulus of elasticity at the matrix temperature, T_m ,
- $\Delta\alpha$ = difference in coefficient of expansion between bead and matrix at temperature $T_g = \alpha_b - \alpha_m$,
- T_g = graphitization temperature,
- α_b = bead coefficient of thermal expansion,
- ΔT = difference between bead temperature and matrix temperature = $T_b - T_m$,
- θ = geometry and stiffness factor.

It is assumed for the purposes of this calculation that the stress-free condition is the fuel graphitization temperature.

A final transverse stress mechanism is the transverse compressive bundling load placed on the fuel elements for the simple purpose of maintaining the core assembly as a more or less integral cylinder. This stress is so small compared with the other stress mechanisms, and the element compressive strength is high enough, that it is neglected.

Turning now to the mechanisms which induce axial stresses in the element, there are internal heat generation, coating-matrix interaction, and bead-matrix interaction, all present in the transverse stress picture.

A major additional contributor is the axial friction stress. Under the tightest controls of flow distribution, fuel loading, etc., there are bound to be element-to-element temperature differences. The calculation of maximum tensile stress in the coolest element in the core is performed according to

$$\sigma_f = E \alpha \Delta T,$$

where

E = modulus of elasticity,
 α = coefficient of thermal expansion,
 ΔT = local interelement temperature difference,

or (whichever gives the lower stress)

$$\sigma_f = \int_x^{52} \mu P_b \cdot \frac{P}{A} dx ,$$

where

x = element station,
 μ = coefficient of friction,
 P_b = bundling pressure,
 $\frac{P}{A}$ = perimeter to cross-sectioned area ratio of fuel element,
 σ_f = axial friction stress.

The stress in the element length over which element-to-element slippage occurs is based on a fuel element without positive axial constraint at the hot end. The stress over the non-slipping, or "locked-up", element length is a simple function of local interelement temperature difference and appropriate properties. Schematically, the axial friction stress varies as shown in Figure 3.

Another axial stress component derives from the temperature difference transversely across the element. Superimposed on the complex thermal pattern within an element (channel walls coolest, element flats hottest) there can be a transverse temperature gradient due to a gross core radial temperature distribution inevitable especially at the core periphery. Assuming a linear gradient across the given element, this stress equals $1/2E\alpha\Delta T$, where ΔT = temperature difference across element flats.

The core pressure drop is the final axial component, but is compressive and is presently neglected to add conservatism to the analysis.

Most of the stresses just described are combined in a two-dimensional (plane strain) finite element structural model (currently 1500 nodes to represent a 1/12 model of the 0.75 inch across flats element) which calculates its own internal temperature distribution and includes such refinements as taking into account the graphite orthotropic properties and the stress-free reference temperature in the coating-matrix interaction computation.

CRITERION OF FAILURE

Having developed, with the complicated model just described, the fuel stress distribution, the next task is prediction of a safety factor or a reliability. Such a prediction depends on defining an adequate criterion of failure. Pos-

sible approaches are: (1) fracture mechanics, (2) Weibull (weakest link) statistics, (3) maximum principal stress, (4) maximum principal strain, and (5) a strain energy criterion.

The fracture mechanics approach¹, with comments on salient advantages and disadvantages, is depicted below.

Definition

failure occurs when

$$\sigma > \sigma_c ,$$

where

$$K_{IC} = Q \sigma_c \sqrt{\pi a} = \text{fracture toughness,}$$

$$Q = \text{geometry factor,}$$

$$a = \text{anticipated flaw size.}$$

Advantages

1. Reasonably well developed technique for selected geometries and stress distributions.
2. Moderate level of testing required.

Disadvantages

1. Of doubtful applicability to material with distributed internal "defects".
2. As-extruded geometry precludes testing of standard geometry fracture mechanics specimens.
3. No obvious method of accounting for volume under load in reliability prediction.

Weibull analysis² is a statistical approach to calculating brittle material failure. In simple terms, the probability of fracture of a component is calculated as a weighted function of the volume under tensile stress and the size of the tensile stresses which are present.

Definition

$$P = 1 - \exp \left[- \int_V \left(\frac{\sigma - \sigma_u}{\sigma_o} \right)^m dV \right] ,$$

where

$$P = \text{probability of fracture,}$$

$$V = \text{volume under tension,}$$

$$\sigma = \text{local tensile stress,}$$

σ_u = minimum material strength,
 σ_o = volume-dependent scale parameter,
 m = scatter parameter;
 (σ_u , σ_o , and m are Weibull constants).

Advantages

1. Directly predicts failure probability including effect of volume under load.
2. Computational method reasonably direct and small extrapolation from current finite element methods.

Disadvantages

1. Requires very extensive testing program.
2. Applicability to graphite not established.
3. Risk of extraneous surface finish effects in small volume specimens.

Maximum principal stress theory is a classic approach to predicting brittle failure when the ultimate tensile strength is locally exceeded. Its potential is summarized as follows.

Definition

Failure occurs when
 maximum tensile stress > ultimate tensile strength.

Advantages

1. Moderate level of testing required.
2. Directly compatible with current finite element methods.

Disadvantages

1. Definition of maximum stress problematic in vicinity of sharp-edged flaws.
2. Probably overconservative with zero allowance for local yielding.
3. No obvious method of accounting for volume under load in reliability prediction.

Maximum principal strain theory has only recently received serious attention as an appropriate failure criterion. Progress to date with this idea does not warrant further discussion here.

A final alternate theory of failure is a strain energy criterion proposed by Andrae³. A prediction of failure based on crack initiation, not crack propagation, is suggested.

Definition

Failure occurs when

$$\frac{1}{A_F} \int_{A_F} \frac{1}{2E} \sigma^2 dA > \frac{1}{2E} \sigma_{ult}^2 ,$$

where

A_F = initial fracture area,

E = modulus of elasticity,

σ = local stress,

σ_{ult} = ultimate tensile strength.

Advantages

1. Moderate level of testing required.
2. Computational method reasonably direct and small extrapolation from current finite element methods.

Disadvantages

1. Lacks adequate experimental verification.
2. No obvious method of accounting for volume under load in reliability prediction.

These theories continue to get our analytic and experimental attention, but a confident choice of a standard failure criterion for the NERVA fuel analysis still seems elusive. In this connection, a notable program to define the graphite failure criterion has been underway in the Oak Ridge National Laboratory Graphite Mechanics group, with much of the work completed under this program being discussed elsewhere during this conference.

RELIABILITY PREDICTIONS

The final comments are a brief discussion of the probabilistic thinking which pervades the NERVA fuel analysis and the interpretation of the analytic results. Excluding the Weibull analysis with its built-in probabilistic failure prediction, the essence of our prediction of probability of failure lies in the so-called "interference theory" or "stress-strength overlap" method⁴. The approach is sketched in Figure 4. The strength curve is generated simply from a probabilistic description of the appropriate material property, ultimate strength, ultimate strain, or fracture toughness, for example. A probabilistic description of the stress is, however, more difficult. The present approach is to use "engineering judgment" on each stress component at the point of maximum combined stress in a manner similar to that shown below. The example below is the calculation of uncertainty in the component of stress due to heat generation:

1. $\sigma_q''' \propto \frac{E\alpha_q'''}{k}$
2. Calculate standard deviation of $(E\alpha_q'''/k)$ from known standard deviations of all parameters by algebra of normal functions.
3. Calculate standard deviation of σ_q''' at point of maximum combined stress by assuming:

$$\frac{\text{standard deviation } (\sigma_q''')}{\text{mean } (\sigma_q''')} = \frac{\text{standard deviation } (E\alpha_q'''/k)}{\text{mean } (E\alpha_q'''/k)}$$

4. Similarly treat all components of stress and combine standard deviations statistically to determine standard deviation of maximum combined stress.

All stress components can be comparably operated upon and then combined statistically to give the failure-governing stress and its standard deviation. The statistical description of the failure-governing stress and of the selected measure of "strength" necessarily involves a statistical description of numerous input parameters. These parameters include fuel geometry, operating environment and fuel material properties. To meet the need of statistically determined properties an extensive material properties test program has been mounted within the NERVA project. The program is unique in covering some exotic materials, in requiring large sample sizes, and in having to take into account extreme radiation and thermal environments as well as exposure to hydrogen and carbon. With the assumption of a normal distribution on both stress and strength, a probability of failure in the component is routinely calculated.

SUMMARY

In summary, the points discussed have been the NERVA fuel element stress mechanisms, the various alternatives available as a criterion of failure, and, finally, the probabilistic schemes present in the analysis. The NERVA fuel element is a highly developed piece of hardware with an impressive history of performance success. However, the complex loads on the element combined with the two-edged demand of even greater performance with increased reliability keep the stress analytic methods stretching current state-of-the-art techniques.

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Fig. 1. NERVA Nuclear Subsystem Cutaway.

Fuel Elements

- SUSTAIN CONTROLLED NUCLEAR HEAT GENERATION
 - PYROCARBON COATED UC_2 FUEL BEADS DISPERSED THROUGH AXM GRAPHITE MATRIX (630 MG/CC MAXIMUM FUEL LOADING)
 - UC IN COMPOSITE WITH ZrC AND GRAPHITE (700 MG/CC MAXIMUM FUEL LOADING)
- LIMIT TOTAL REACTIVITY LOSS TO \$1.00 AT END OF LIFE
 - CARBIDE COATING OF FLOW CHANNELS
- AFFORD HEAT TRANSFER FROM FUEL ELEMENT TO H_2 PROPELLANT
 - 19 FLOW CHANNELS IN EACH $3/4$ INCH HEX, x 52 INCH LONG FUEL ELEMENT

Fig. 2. NERVA Fuel Element Description.

Fig. 3. Axial Friction Stress Schematic.

Fig. 4. Schematic of Overlap Method of Failure Probability Prediction.

V R

X THE NERVA NUCLEAR SUBSYSTEM
FRICTION-DAMPED LATERAL SUPPORT SYSTEM

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ABSTRACT

The severe environmental and operational requirements for the NERVA engine have led to the extensive use of graphite in the lateral structural support system (LSS) of the reactor core assembly. The demanding LSS requirements include the condition that the LSS must limit the radial load on the reactor core between narrow limits while permitting thermal and hydraulic motions. At the same time it must limit or dampen any core dynamic motions which are induced during the various phases of life of a space vehicle. The novel feature in the LSS design is the use of the wear properties of graphite, in particular, the controlled friction properties of graphite, to control and limit the core assembly static and dynamic lateral loads. The evolution of the LSS from those of the early KIWI series reactors to the present design concept is traced.

A model of a base excited, Coulomb-damped single degree of freedom system is used to investigate the first mode dynamic response. Den Hartog has found the exact solution to this problem for the case when sliding occurs over each full half-cycle and has limited his attention to only an approximate solution for the case of stop motion, during which the exciting base and the excited mass are locked together for a portion of the cycle. Because of space limitations in the NERVA design, however, the stop motion case is found to be the limiting case dynamically.

Dimensionless curves describing the overall motions are presented and used to discuss the role of graphite friction in the LSS design.

NOTE: The Nuclear Engine for Rocket Vehicle Application Program (NERVA) is administered by the Space Nuclear Propulsion Office, a joint office of the U.S. Atomic Energy Commission and the National Aeronautics and Space Administration. Aerojet Nuclear Systems Company as prime contractor for the engine system and Westinghouse Electric Corporation as subcontractor for the nuclear subsystem, are developing a nuclear propulsion system for space applications.

INTRODUCTION

The structural design of a nuclear reactor for space operation poses some interesting design problems. The reactor must not only withstand the high temperature and high radiation levels typically encountered in a high power reactor operation, but it also must be capable of multiple restarts and long life without degradation of its structural reliability. The reactor is also subjected to a broad range of dynamically induced environments, such as launch and space operation vibrations, and the varied space natural environments including an ultra high vacuum. The purpose of this paper is to discuss the use of graphite in the structural design of a particular component, the core assembly lateral support system of the NERVA Nuclear Rocket Engine.

ENVIRONMENT

The severe environmental and operational requirements for the NERVA engine have led to extensive use of graphite in the lateral support system (LSS) of the reactor core assembly. The demanding LSS requirements include the requirement that the LSS must limit the radial loads on the reactor core between narrow limits while permitting thermally and hydraulically induced radial motions of almost 1/2 inch. While the lower limit on the core radial load is imposed to bundle the core elements in order to minimize the possibility of the occurrence of any localized core motions, the upper load limit is imposed to limit the axial friction stress in the core assembly elements as they inexorably expand when subjected to temperatures approaching 4500°R. In conjunction with these thermal and hydraulic requirements, the LSS must also limit or dampen any reactor dynamic motion in order to attempt to isolate the core from the various dynamic loads induced during the various phases of life of a space vehicle. Among difficulties imposed here is the fact that the various dynamic requirements span broad frequency ranges from the very low frequencies below 35 hz encountered in the launch of a Saturn V type vehicle to the nuclear stage space operation vibration spectrum which is rich in the intermediate to high frequencies from 100 hz to 2 khz.

The LSS, consequently, must be sufficiently flexible to permit relatively large thermal motions, yet it must be stiff enough to limit or dampen vibratory motions over a broad frequency range. These manifold requirements have led in the past to the use of the wear properties of graphite, in particular, the controlled friction properties of graphite to control and limit the core assembly static and dynamic lateral loads.

DESIGN HISTORY AND STATIC ANALYSIS

The basic need for a LSS was discovered early in the nuclear engine development program when one of the KIWI reactor tests was terminated at the Los Alamos Scientific Laboratory because of extensive structural failures. The basic problem was traced to the lack of adequate radial bundling which led to the occurrence of uncontrolled flow induced vibrations. From that point onwards in the development program, the question turned from one of whether to mechanically bundle the core to one of how to bundle the core assembly.

An early design used by the Westinghouse Astronuclear Laboratory (WANL) in the technology program to develop the NERVA reactor is shown in Figure 1. In this basic design, typical of the NRX-A through NRX-A5 reactors and of the experimental XE-1 and XE-2

reactors, a preload force is applied by springs which are attached to the inner graphite reflector, through graphite plungers which in turn transmit the force to the graphite load rings. One of the criteria for choosing the spring preload force is that the LSS provide sufficient radial force to bundle the core elements. The inner load ring is positively seated against the outer ring. As the core assembly expands and contracts radially in its thermal cycles, the load rings slide on each other with the friction increasing the net bundling force during outward core motion and decreasing the net bundling force during core contraction. Because of the radial thermal symmetry in the reactor, each plunger in an axial row is subjected to approximately the same conditions.

While thermal motions tend to be radially symmetric, dynamic motions tend to be unidirectional. As the reacting friction forces oppose dynamic motions, the resultant reacting forces act opposite to the direction of motion with a maximum reacting force equal to $G_{BA} W$, where W is the effective core assembly weight and G_{BA} (so called "break away" acceleration) is the input G level at which relative motion would first occur between the core assembly and the reflector. Hence the parameter G_{BA} is a measure of the frictional forces present in the design.

In this design then, graphite friction is used to eliminate any relative dynamic motions between the core assembly and the reflector assembly for reflector G levels less than G_{BA} , to dampen relative motions for dynamic levels above G_{BA} and to aid in providing positive bundling to the core assembly.

A criterion for the design of the last of the NERVA technology reactors, the NRX-A6 reactor, was that the design had to be capable of extension to the larger R series reactors with its more stringent requirements. The inner graphite reflector of the early LSS designs was made from a single cylinder of graphite. At that time, a suitable extrusion of structural graphite of the size required for the larger R series reactors could not be obtained. Obviously a new LSS was required to meet the general criterion for the NRX-A6 design. WANL decided to utilize much of the experience gained during the successful LSS operation in the previous NRX reactors. The basic design used in the NRX-A6 reactor is shown in Figure 2.

In this design, coil springs are used to load the plungers which extend into the metallic reflector. The outer load ring was canted to effectively multiply the spring force with wedging action. Hence, the role of graphite friction was increased in this design. It should be noted that the basic lateral support designs of the technology series reactors have undergone eight series of reactor full power and mapping tests and also numerous simulated vibration tests without a structural failure of any graphite part.

Since the NRX-A6 design, the requirements for LSS radial displacements have increased by more than 91 percent while the effective core weight has increased by more than 116 percent. These increases were in large part caused by fuel structural modifications as discussed in another paper presented at this conference. But, of course, the decrease in stiffness and the increase in friction induced by these requirements cannot be obtained freely without any structural penalties.

Consider, for example, the effects of graphite friction shown in Figures 3, 4, and 5 in which are shown results for the current LSS reference design and two perturbations of this design. In order to understand the contributions of each of the components to the total friction of the system, only the load ring bearing angles, the plunger face angle and the coefficient of friction were considered as variables. Manjoine¹ has shown that the friction coefficient in air

of graphite on the metals considered for the LSS design approaches that of graphite on graphite so that only one friction coefficient need be used for the system. Each of the two angles θ_1 and θ_2 shown in Figure 2 were varied in turn by 10° with the results shown in Figures 3 and 4 for the total system and in Figure 5 for the plunger effects alone.

One should note that an increase in θ_2 from 60° to 70° has no effect on pin bending stress parameter $\sigma_p / \sigma_{p\mu=0}$ shown in Figure 5, but it does show a small reduction in the dynamic parameter G_{BA} and the bundling parameter $p/p_{\mu=0}$ shown in Figures 3 and 4, respectively. For these ring angles, the friction interfaces between the two load rings and between the inner load ring and the core assembly contribute only a small amount to the total friction effects on the LSS. Plunger friction, on the other hand, contributes the pre-dominant share of the friction effects of the LSS. A change in the inner load ring and plunger angle from 30° to 20° reduces the parameter G_{BA} by approximately 70 percent from that value for the reference design and for a coefficient of friction of .3 as shown in Figure 3.

The vertical asymptotes in Figures 3, 4 and 5 represent absolute limits of the use of friction in the design for small motions about the installed position. At these limits the friction system becomes "perfectly efficient" and binding of the component parts occurs. A comparison of Figures 3, 4 and 5 shows that for motion impending in a radially outward direction on this design, binding apparently first occurs on the plunger. The graphite plunger, then, is the key structural element in the present LSS design. It is subjected to a combination of spring loads, the guide loads at two interfaces in the reflector and the wedge loads at the inner load ring interface.

Let us look further at the stress in a single plunger. The maximum bending stress in the plunger occurs at the location where the plunger pin is guided in the reflector. A free body diagram of the plunger shown in Figure 2 can be used to obtain the following equation for the maximum plunger bending stress for the case of motion impending in a radially outward direction.

$$\sigma_p = \frac{32k}{\pi D_p^3} (S_o + X)(L_1 - X) A/B \quad (1)$$

where

$$A = (L - L_1 + X - \mu/2 (D_h - D_p)) (.5 + .866\mu)$$

$$B = \frac{.866 (L - L_1 + X) - \mu (1.5L + .433 (D_h - D_p)) + \mu^2 (.5 D_h - .866 (L + L_1 - X)) - .433 \mu^3}{(D_h + D_p)}$$

- k is the spring constant of the compression spring
- μ is the uniform coefficient of friction of the graphites
- S_o is the preload deflection of the spring
- D_h is the diameter of the plunger head
- D_p is the plunger diameter
- L_1 is the installed plunger overhand length inside of the reflector
- X is the outward plunger displacement measured from the installed position

From Eq. (1), we note that binding occurs when the term B vanishes and therefore depends only on the geometrical and friction parameters of the system and not on the applied loads. Furthermore, the lowest critical value of the coefficient of friction for plunger binding is obtained when the displacement X reaches its maximum negative value. This condition can only occur if the core assembly shifts as a rigid body to one side of the reflector and attempts to return to its original position. For the current reference designs such a possibility can only

occur in the case of full amplitude dynamic motions which are discussed more fully in another section.

The displacement X_1 corresponding to the maximum bending stress depends on the value of μ . It is approximately zero for small values of μ and increases negatively as μ increases until it reaches the limit discussed above. For a typical design the nominal value of the maximum plunger bending stress is approximately 3000 psi with a 99 percent reliability at a 95 percent confidence level that the value will not exceed 4800 psi. For these small graphite parts, this represents a considerable stress level.

There are 756 plungers, each approximately 3 inches long with an average diameter of almost 1/2 inch used in 21 axial rows in the current reference design to support the core assembly. All of these plungers, of course, are not subjected to the same loads. Dynamic motions tend to be unidirectional and produce nearly identical conditions for the plungers in line with the motion at each load ring row, while thermal motions tend to be radially symmetric at each row with a significant axial row to row variation. Hence, 21 of the 756 plungers reach their maximum stress for a constant μ at any time under dynamic motions while 36 plungers are at a maximum stress condition for plungers subjected to thermal motions. Because of the wide spreads in the plunger stress and strength distributions caused chiefly by the large number of pins subjected to varying stress conditions and also by the variation in μ a probabilistic approach can be used to compute the LSS structural reliability.

The plungers are made of PO-3 graphite, which is a fine grain molded structural graphite. The material is nearly isotropic in its strength distribution with a bending strength value at a 99 percent reliability with a 95 percent confidence level of over 6100 psi in the temperature range of interest. The high density and small coke particle size of PO-3 graphite help to obtain a good surface finish, resistance to abrasion, and close dimensional tolerances on hardware components. The material has been successfully used in the varying environments encountered in the NRX series of NERVA reactors.

The NERVA Engine must also withstand the natural environment encountered in space without degradation of its performance characteristics or its structural reliability. In this "space soak" condition, the LSS could be subjected to a temperature range of approximately 400°R to 900°R in a vacuum below 10^{-9} torr. Because natural graphite is known² to have a higher friction coefficient in an ultra high vacuum and because these temperature extremes would naturally induce a tendency for LSS thermal motions, friction effects may be required to be reduced in a space environment for the NERVA design.

WANL has initiated a large scale test program in order to determine the environmental effects on the coefficient of friction of the LSS materials. In this test program, the static and dynamic coefficients of friction of the various graphite and graphite metal combinations, presently used or considered as candidates, will be tested over a broad temperature range in air and in a 10^{-9} torr vacuum. The effects of moisture content in air, nitrogen and hydrogen gases will also be determined. This is one of a number of materials programs initiated at WANL with the long range goal to estimate the statistical values of the test parameters as well as average values. One of the materials to be tested is P-03N which is an impregnated graphite that is reported to have a low friction coefficient in a vacuum. We feel that our program to determine the effects of the environment on the LSS materials along with our plans to test various methods of controlling the coefficient of friction, such as suitable impregnations and coatings, offers the best hope of obtaining a LSS of high reliability for the NERVA Engine.

DYNAMIC ANALYSIS

The predominant use of graphite friction in the LSS design is to dampen any relative motions between the reflector and the core assembly for reflector input G levels greater than G_{BA} . We have previously mentioned that the LSS must have flexibility to accommodate core assembly and reflector thermal and hydraulic displacements while ensuring sufficient core bundling. This introduction of flexibility into the LSS design invites the possibility of relative dynamic motions for sufficiently high G levels. The results of Figures 3 and 5 show, however, that an LSS design with relatively large amount of friction, that is, with a high value of G_{BA} , may also encounter relatively high plunger pin bending stresses and may not be adequate in a space soak environment. It appears that an efficient LSS design must allow and account for relative dynamic motions for high G levels.

Only preliminary estimates of the dynamic environment on the reflector are available at this time because the Reusable Nuclear Stage design is only in the preliminary stage of development. We expect the launch condition with a Saturn V type vehicle to present the most severe dynamic condition for the LSS with a $\pm 3G$ reflector sinusoidal acceleration input possible over a range from 5 to 35 hz. Taking into consideration the input displacement attendant to a $3G$ level at an LSS first natural frequency of approximately 10 hz together with the imposed space limitations of about 0.5 inch of allowable motion, one concludes that very little dynamic magnification can be allowed in the low frequency ranges.

The basic use of friction in the dynamic design of the LSS can be determined by examining the first mode dynamic response of the LSS and neglecting for our purposes any additional restraint at the forward end of the core assembly. Let us consider then a base harmonically excited single degree of freedom system with Coulomb damping.

The forced response of such a system has been considered by Den Hartog^{3,4} who, however, principally considered the absolute motion of the excited mass and derived an exact solution only for the case when slipping occurred over each half cycle of motion. Due to the space limitations of the LSS, however, we must also consider the region of "stop" motion in which slipping occurs over only a portion of each cycle.

The condition for stop motion is

$$|M \ddot{X} + K (X - X_1)| \leq P_s \quad (2)$$

and for relative motion

$$M \ddot{X} + K (X - X_1) + P_k \text{Sgn} (\dot{X} - \dot{X}_1) = 0 \quad (3)$$

where

- M is the excited mass
- K is the spring constant
- P_s is the static friction force
- P_k is the kinetic friction force
- X and X_1 are the coordinates of the excited mass and base respectively
- Sgn is the sign function

In the above equations, dots over a variable denote time derivatives. Introducing the dimensionless variables $\bar{X} = X/X_1$, $\bar{Y} = \bar{X} - 1$, $\bar{t} = \omega_n t$, where ω_n is the natural frequency of the equivalent single degree of freedom system, into Eq. (2) and (3), we obtain the dimensionless analogues to Eq. (2) and (3)

$$\left| \ddot{\bar{X}} + \ddot{\bar{Y}} \right| \leq \alpha_s \quad (2a)$$

$$\ddot{\bar{X}} + \ddot{\bar{Y}} + \alpha_k \text{Sgn } \dot{\bar{Y}} = \sin \beta \bar{t} \quad (3a)$$

where dots over a variable now denote differentiations with respect to the variable $\bar{t}$ and the dimensionless force and frequency ratios are defined by

$$\begin{aligned} \alpha_s &= P_s / (K X_1) \\ \alpha_k &= P_k / (K X_1) \\ \beta &= \omega / \omega_n \end{aligned} \quad (4)$$

For our purpose here, we can assume as a first approximation $\alpha_s \approx \alpha_k = \alpha$ so that in the remainder of this paper we can drop the subscript on α . This assumption is equivalent to assuming that the value of kinetic friction coefficient is sufficiently close to the static coefficient of friction for graphites.

For harmonic base motion, it follows from Eq. (2a) and (4) that sliding motion occurs in any cycle when $\alpha = \beta^2$. For a constant harmonic displacement input, the above condition defines the frequency ratio separating the region of locked-up motion from that of sliding motion. For a constant harmonic acceleration input, the condition defines the breakaway G level G_{BA} .

The solutions of Eq. (2a) and (3a) for the locked and sliding phases, respectively, are:

$$\bar{X} = 1 + \bar{Y}_0 \quad (5a)$$

and

$$\begin{aligned} \bar{X} &= (\bar{X}_0 + \text{Sgn } \dot{\bar{Y}}_0 - \frac{1}{(1-\beta^2)} \sin \beta \bar{t}_0) \cos (\bar{t} - \bar{t}_0) \\ &+ (\bar{X}_0 - \frac{\beta}{(1-\beta^2)} \sin \beta \bar{t}_0) \sin (\bar{t} - \bar{t}_0) \\ &- \alpha \text{Sgn } (\dot{\bar{Y}}) + \frac{\sin \beta \bar{t}}{(1-\beta^2)} \end{aligned} \quad (5b)$$

where the dimensionless initialized time is defined by $\bar{t}_0 = \omega_n t_0$ and the subscript o refers to conditions at the time of initiation of sliding motion.

Solutions to Eq. (5a) and (5b) have been obtained with the aid of a CDC-6600 digital computer for various values of the parameters α and β and are shown in Figure 6 for the dimensionless relative motion parameter $\bar{Y}$ and in Figure 7 for the peak dimensionless acceleration of the excited mass $\bar{G} = \ddot{X}/X_1$. It is apparent from these figures that the parameters $\bar{Y}$ and $\bar{G}$

are unbounded at $\beta = 1$ for values $\alpha < \pi/4$. This well known feature of a Coulomb friction damped system is a consequence of the fact that the damping force is independent of the driving amplitude. As β increases without bounds, the motion becomes seismic in which case $\overline{Y} \rightarrow 1$ and $\overline{G} \rightarrow 0$. For values of β greater than approximately $\sqrt{2}$, the use of graphite friction in the LSS system design serves to isolate the core assembly from first mode response to imposed external vibrations.

Because Den Hartog^{3,4} previously considered only the absolute motion of the excited mass and because the phase angle relations for the stop motion case have not yet been determined, it is not possible to make a direct comparison between the results of Den Hartog and the results of Figure 6. The results of Figure 6, however, compare favorably with the limited results of analog computer studies⁵ for the same problem. The results of Figure 7 are new and are presented here for the first time.

It is observed from the data of Figures 6 and 7 that stop motion changes the basic character of the forced response of a single degree of freedom system at frequencies removed from the natural frequency and adds additional complexities to the modal response of the LSS.

CONCLUSIONS

Graphite has been extensively used in the NERVA lateral support system. In particular, graphite friction has been used to protect the core assembly from a broad spectrum of dynamic loads and to aid in providing positive core element bundling. The effects of friction, however, increase the stress in some LSS components and may lead to potential binding problems, especially in an ultra high vacuum. For this reason, LSS designs of the same form as those used in the NERVA technology series reactors may not necessarily be the reference design for future NERVA reactors. It appears likely, however, that the future LSS designs for the NERVA engine will be based on many of the features of previous designs discussed in this paper because of the successful test history of previous reactors.

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Fig. 1. The NRX-A5 Reactor Lateral Support System Design.

Fig. 2. The NRX-A6 Reactor Lateral Support System Design.

Fig. 3. Friction Effects on G_{BA} for Various Load Ring Angles.

Fig. 4. Friction Effects on Bundling Pressure for Various Load Ring Angles.

Fig. 5. Friction Effects on Plunger Stress for Various Load Ring Angles.

Fig. 6. Amplitude Ratios for Base Excited Vibrations with Coulomb Damping.

Fig. 7. Acceleration Ratios for Base Excited Vibrations with Coulomb Damping.

STEEL-TO-GRAPHITE CONNECTIONS

ANALYZED BY FINITE ELEMENT TECHNIQUES

FOR USE IN NERVA NOZZLE EXTENSION

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ABSTRACT

The selection of fibrous reinforced graphite for the NERVA** rocket nozzle extension was based on a study of various alternate materials. A major problem in the design of the nozzle extension was the structural analysis of the connection of a graphite flange, heated by hot exhaust gases, to a steel flange, which is simultaneously cooled by liquid hydrogen and heated by nuclear radiation. This type of problem is readily solved by the application of a finite-element program for cylindrically orthotropic solids of revolution. The design of the

joint is then an application of the full potential of the finite-element method. The connection is modeled for finite-element stress analysis and an arbitrary temperature distribution, determined by a finite-element thermal analysis, is applied. The objectives for the final design concept were to achieve, as nearly as possible, the smallest principal stresses in the plane of the cross section, and uniform hoop compression. The treatment of the bolts and seals in the finite-element model is described.

I. INTRODUCTION

The NERVA engine system, as now planned, will be a 75,000-lb-thrust nuclear rocket engine to be used for deep-space travel. The engine will use a high expansion nozzle with a throat-diameter to exit-diameter ratio of 1 to 10, and with an extension which expands the hot exhaust gases from an area ratio of 24 to 1 to an exit ratio of 100 to 1. Stainless steel, aluminum, and fibrous-reinforced graphite were investigated for possible use in the nozzle extension. The steel and aluminum extensions required cooling to operate in the radiation environment, while the graphite extension did not require cooling. The cooling requirements added penalties

both for weight and for system performance; for this reason, the fibrous reinforced graphite was selected as the candidate material.

Coincident with the system perturbations, each concept presented problems. For example, the stainless steel extension had to use a tubular construction, and the joint between the nozzle and the extension would experience a severe thermal discontinuity (of the order of 1000°F).

The graphite extension presents a similar problem, as discussed in this paper.

II. NOMENCLATURE

α_i = coefficient of thermal expansion for cylinder i , in./in. per degree

ΔT_i = temperature change from ambient temperature for cylinder i , degrees

R = radius of cylinder, in.

M_i = internal moment on edge of cylinder at location i , in.-lb/in.

V_i = internal shear on edge of cylinder at location i , lb/in.

λ_i = damping factor for cylinder i , equal to $\frac{4}{\sqrt{3(1-\nu^2)}} / \sqrt{Rt}$ for isotropic material

D_i = flexural rigidity of cylinder i , equal to $Et^3/12(1-\nu^2)$ for isotropic cylinder

η = ratio of stiffnesses of two adjoining cylinders; for equal thickness this is equal to ratio of the Youngs Moduli

l_i = length of cylinder i , in.

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III. THE PROBLEM

The nozzle extension is joined to the nozzle at an area ratio of 24:1. The major source of loading at the joint will be the result of the temperature difference between the two parts. The stainless steel nozzle is heated by nuclear radiation and hot exhaust gases, and cooled by liquid hydrogen. The nozzle extension is heated by the hot exhaust gases and uncooled.

Two typical design concepts are shown in

Figures 1 and 2, illustrating the problem of determining the stresses in the two parts and recommending efficient design changes if those stresses are too high.

We then are faced with determination of thermal stresses in the mechanical joint of a steel flange and an orthotropic graphite flange with widely varying temperature distributions.

NOZZLE/GRAPHITE COMPOSITE SKIRT EXTENSION INTERFACE CONCEPT #4

NOZZLE/GRAPHITE COMPOSITE SKIRT EXTENSION INTERFACE CONCEPT #9

IV. A SIMPLIFIED APPROACH

This problem can be reduced to its simplest form if all factors but material and temperature differences are ignored, and the nozzle extension considered as a simple cylinder of steel and graphite joined at a location "A" by an assumed perfect bond. Let us further assume that the steel is heated uniformly for its entire length, that the graphite is heated uniformly for its entire length with a resulting step change in temperature at "A" (see Figure 3), and that both the steel and graphite are isotropic and of equal thickness.

Because of the difference in temperature, there will be a difference in the free thermal displacement of the two cylinders. The bond between the two can only exist in the presence of a local shear and bending moment at A. The shear and moment are assumed to act as shown in Figure 4.

If we specify that the displacement and rotation at Point A be equal for both cylinders, we find that these equations must be satisfied:

$$R\alpha_1 \Delta T_1 + \frac{M}{2\lambda_1^2 D_1} - \frac{V}{2\lambda_1^3 D_1} = R\alpha_2 \Delta T_2 + \frac{V}{2\lambda_2^3 D_2} + \frac{M}{2\lambda_2^2 D_2} \quad (1)$$

$$-\frac{M}{\lambda_1 D_1} + \frac{V}{2\lambda_1^2 D_1} = \frac{V}{2\lambda_2^2 D_2} + \frac{M}{\lambda_2 D_2} \quad (2)$$

From these equations we can solve for the interface shear, V , and moment, M , in terms of a modulus ratio, η , in which

$$\eta = \frac{D_1}{D_2} = \frac{E_1}{E_2} \quad (3)$$

SIMPLIFIED MODEL

FIGURE 3

FREE-BODY DIAGRAM CASE 1

FIGURE 4

and finally

$$V = 4\lambda^3 D_1 R(\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta + 1}{\eta^2 + 6\eta + 1} \quad (4)$$

$$= 4\lambda^3 D_2 R(\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta(\eta + 1)}{\eta^2 + 6\eta + 1}$$

$$M = -2\lambda^2 D_1 R(\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta - 1}{\eta^2 + 6\eta + 1} \quad (5)$$

$$= -2\lambda^2 D_2 R(\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta(\eta - 1)}{\eta^2 + 6\eta + 1}$$

where

λ = damping constant for the cylinder,

D = flexural rigidity of the cross-section

Based upon the foregoing, we can also find the deflection, δ , and the rotation, θ , at the joint:

$$\delta = \frac{R_1 \alpha_1 \Delta T_1 \eta(\eta + 3) + R_2 \alpha_2 \Delta T_2 (3\eta + 1)}{\eta^2 + 6\eta + 1} \quad (6)$$

$$\theta = \frac{R \alpha_1 \Delta T_1 4\lambda \eta - R \alpha_2 \Delta T_2 4\lambda \eta}{\eta^2 + 6\eta + 1} \quad (7)$$

The following expressions for the hoop stress resultants can now be derived, as:

$$N\theta_1 = -t_1 E_1 (\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{(3\eta + 1)}{\eta^2 + 6\eta + 1} \quad (8)$$

$$= -t_2 E_2 (\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta(3\eta + 1)}{\eta^2 + 6\eta + 1}$$

$$N\theta_2 = t_1 E_1 (\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta + 3}{\eta^2 + 6\eta + 1} \quad (9)$$

$$= t_2 E_2 (\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \frac{\eta(\eta + 3)}{\eta^2 + 6\eta + 1}$$

where

t = cylinder thickness

E = Young's Modulus of the material

These expressions are plotted in Figures 5 and 6 as functions of η , the ordinate and abscissa being dimensionless. The expressions adjacent to the curves represent the forces divided by the fixed-end-forces and the radial expansion difference. The fixed-end-forces are those forces which result from a clamped-edge cylinder subjected to a unit radial displacement.

A better representation of the problem could be to include a thickened area near the joint at Location A. The problem can now be idealized as two long cylinders joined through two shorter cylindrical segments, as shown in Figure 7. The subscripts 1 through 4 indicate the different parts of the idealized structure, reading from left to right in the figure. The subscripts on the forces correspond to the points, reading from left to right, at which the structure was cut for analytic purposes with the forces shown as assumed.

INTERNAL FORCE DISTRIBUTION - CASE 1

FIGURE 5

INTERNAL FORCE DISTRIBUTION - CASE 1

FIGURE 6

FREE-BODY DIAGRAM CASE 2

FIGURE 7

The solution of this problem is governed by compatibility equations similar to those of the previous problem, but now, rather than two unknown forces, we have six unknown forces, so the equations can more readily be expressed in matrix form as:

$$\begin{bmatrix} (-F_{11} - F_{12}) & (F_{31} - F_{32}) & (F_{22}) & (F_{42}) & 0 & 0 \\ (F_{31} - F_{32}) & (-F_{51} - F_{52}) & (-F_{42}) & (F_{62}) & 0 & 0 \\ (F_{22}) & (-F_{42}) & (-F_{12} - F_{13}) & (F_{32} - F_{33}) & (F_{23}) & (F_{43}) \\ (F_{42}) & (F_{62}) & (F_{32} - F_{33}) & (-F_{52} - F_{53}) & (-F_{43}) & (F_{63}) \\ 0 & 0 & (F_{23}) & (-F_{43}) & (-F_{13} - F_{14}) & (F_{33} - F_{34}) \\ 0 & 0 & (F_{43}) & (F_{63}) & (F_{33} - F_{34}) & (-F_{53} - F_{54}) \end{bmatrix} \begin{bmatrix} V_1 \\ M_1 \\ V_2 \\ M_2 \\ V_3 \\ M_3 \end{bmatrix} = \begin{bmatrix} -R(\alpha_1 \Delta T_1 - \alpha_2 \Delta T_2) \\ 0 \\ -R(\alpha_2 \Delta T_2 - \alpha_3 \Delta T_3) \\ 0 \\ -R(\alpha_3 \Delta T_3 - \alpha_4 \Delta T_4) \\ 0 \end{bmatrix} \quad (10)$$

in which

$$\left. \begin{aligned} K_1 F_{11} &= 2\lambda_i (\sinh \lambda_i l_i \cosh \lambda_i l_i - \sin \lambda_i l_i \cos \lambda_i l_i) \\ K_1 F_{21} &= 2\lambda_i (\sinh \lambda_i l_i \cos \lambda_i l_i - \sin \lambda_i l_i \cosh \lambda_i l_i) \\ K_1 F_{31} &= 2\lambda_i^2 (\sinh^2 \lambda_i l_i + \sin^2 \lambda_i l_i) \\ K_1 F_{41} &= 4\lambda_i^2 (\sinh \lambda_i l_i \sin \lambda_i l_i) \\ K_1 F_{51} &= 4\lambda_i^3 (\sinh \lambda_i l_i \cosh \lambda_i l_i + \sin \lambda_i l_i \cos \lambda_i l_i) \\ K_1 F_{61} &= 4\lambda_i^3 (\sinh \lambda_i l_i \cos \lambda_i l_i + \sin \lambda_i l_i \cosh \lambda_i l_i) \end{aligned} \right\} \quad (11)$$

and

$$K_1 = \frac{E_i t_i}{R^2} (\sinh^2 \lambda_i l_i - \sin^2 \lambda_i l_i) \quad (12)$$

where

i = different parts of the structure from Figure 7.

These equations are not solved as readily as are equations (1) and (2); however, they can be evaluated numerically for several cases and the resulting forces can be expressed in non-dimensional form, as in Figures 5 and 6, as fractions of the fixed-end-forces. These results are shown in Figures 8 and 9. All variables except the thickness and length of Sections 2 and 3 were held constant or reduced to unity, and the step change in thermal displacement was enforced between Sections 2 and 3. The ratio, η , now indicates the ratio of the Young's Moduli.

In both of the above examples, assumptions were made to provide results in terms of the most significant variables. Addition of more variables would further complicate the results, and defeat the purpose of those examples.

The results have shown that, for the simplest case, the internal load distribution in two cylinders with dissimilar materials and a step change in temperature can be expressed as a fraction of the fixed-end-forces on the cylinders due to that temperature difference. The fraction is a function of the stiffness (modulus) ratio of the two cylinders.

An extension of the simple case to include local stiffening showed essentially the same results; however, in the case of local stiffening the fraction can be reduced significantly. At high modulus ratios the fraction approaches a constant value, making the internal load distribution uniquely temperature dependent.

V. THE NERVA NOZZLE EXTENSION

Since the problem has now been characterized in its simplest forms, let us consider a real case. There are significant differences between the simple problems discussed in the previous paragraphs and the true article.

The steel nozzle exit area is a jacketed U-tube conical segment with a toroidal coolant manifold near the exit, and the nozzle extension is a conical shell of orthotropic graphite material. So the hot exhaust gases can be contained, the two are bolted together with a steel lug and bolt attachment with Grafoil* or Pyrolytic graphite foil, being used as the sealing material. Grafoil is generally orthotropic, with the principal axes of orthotropy at an angle to the principal axes of the graphite orthotropy.

The two sections are heated by the hot exhaust gases; the steel areas are also heated by nuclear radiation. One can easily see that the derivation of design equations, similar to those of the two simple examples, would be very difficult to determine. In addition, when consideration is given to the temperature distributions in the two previously cited design concepts (shown below in Figures 10 and 11) there is a serious question of whether or not stress analysis would be possible to any degree of accuracy, were it not for recent advances in the field of numerical stress analysis.

TEMPERATURE °R PROFILE NOZZLE/SKIRT EXTENSION INTERFACE CONCEPT #4

FIGURE 10

TEMPERATURE °R PROFILE NOZZLE/SKIRT EXTENSION INTERFACE CONCEPT #9

FIGURE 11

Considering, then, only the problem of the stress analysis, we can use to great advantage a finite element program written for axisymmetric solids of revolution with cylindrical anisotropy (orthotropy). The finite element methods have been covered in great detail in recent literature and will not be discussed here, except to say that the input to the program includes material orthotropy relative to arbitrary local material axes for an element and a transformation angle between these local axes and the global axes. In the construction of the system stiffness matrix, the program performs the tedious quadratic trigonometric transformation of material stiffnesses which then results in the orthotropic stiffnesses in the global system.

A typical finite element model is shown in Figure 12, below. Through application of the appropriate temperatures to this model, we can readily determine the stresses in both parts and recommend changes, if needed, which follow the results of the simplified approaches, above.

FIGURE 12

It should be noted that the bolts and lugs are included as continuum in the finite-element model. Since the program formulation includes material orthotropy, the bolt "material" is fictitious and is intended to represent the flexibility of the real bolts. The bolt and lug materials occupy the same space but are not interconnected. The two are joined at points of contact only, and as a result the bolt material goes "through" the lug material in much the same manner as does the real bolt.

Note also that the sliding surfaces are rigidly connected. This is a close approximation for bolted surfaces if the frictional forces developed are high.

The two example cases may now be reconsidered in light of the discussion of the NERVA nozzle. The example cases showed that the internal force distribution, and therefore the internal stresses, were simply functions of the thermal displacement difference and a fraction depending upon the modulus ratio of the two parts. In addition, the fraction tended to become stationary at high modulus ratios.

*Trademark, Union Carbide Co.

In the NERVA nozzle extension joint, in addition to the inherently separate moduli of the two materials, thermodynamic engine cycle demands control the coolant manifold size, and the thickness of the graphite is partially controlled by radial thermal gradients. There was for this reason, only a minimal chance that the stiffness of the joint could be balanced.

The one apparent possibility was to match the radial growth of the two parts by allowing non-uniform heating of the two in inverse proportion to the coefficients of thermal expansion. This was possible because of the finite-element program (discussed above) which allows an accurate determination of the growth of the two parts. The adopted design philosophy was based on a trial-and-error solution, which successively treated a new design based on the most previously established design. Changes in design which improved the problem were adopted, when possible, and changes which worsened the situation were noted for future reference.

The work, to date, has primarily resulted in gross dimensions and stresses to an order of magnitude with these major findings:

- 1) The first concept, without a coolant manifold and with contact between steel and graphite, demonstrated extremely high stresses in both steel and graphite. The result was to isolate the graphite and cool the steel.

- 2) The presence of coolant near the aft nozzle flange had the desired effect on the

graphite stresses, but the steel flange tended to heat locally, thus creating high fatigue stresses in the steel.

- 3) Moving the coolant torus on top of the nozzle aft flange had the desired effect. The deflection of the steel flange (due primarily to rotation of the torus and flange assembly) and the deflection of the graphite flange were nearly equal, with resulting low discontinuity stresses. The internal stresses due to the axial and radial thermal gradients were small enough to establish the feasibility of the joint.

- 4) Attempts to balance the thermal displacements have shown that the steel geometry predominates in the design. Major changes in the graphite flange are overshadowed by minor changes in the steel geometry.

- 5) The graphite stresses are dependent on the stiffness of the "Grafoil". Thick, flexible sections of Grafoil tend to isolate the graphite flange, but the material and frictional properties are not fully known at this time.

The next step will be detail design of the nozzle joint, which will use the same trial-and-error method as previously mentioned, but will be primarily concerned with changes on a much smaller scale. This detail design will have the objective of effecting changes to the degree that the flange will be operating at steady-state conditions with (as nearly as possible) uniform triaxial compressions of small magnitude. Small triaxial compressive stresses will enhance the reliability of the flange at steady-state operation.

VI. CONCLUDING REMARKS

While the summary above has presented the results of the preliminary work on the bolted joint of the Nozzle-to-Nozzle Extension for the NERVA engine, still a large amount of work remains to be done to finalize the design. The final phases will analyze transient thermal stresses and fatigue stresses in addition to the steady-state loads discussed above.

Upon completion, the NERVA Nozzle Extension will be a conical shell with diameters varying from 5 to 10 ft, and slightly less than 12 ft in length, and will weigh approximately 600 lb. The entire shell must be carried by the forward joint and as a result the first few inches of the extension will receive a great deal of attention.

The use of a finite-element program has lent a degree of accuracy to the development of the conceptual design which was not possible a few years ago. The major problems in the analysis, that is, the joining of two widely dissimilar materials with widely varying and non-uniform temperature distributions are handled readily by this program. It is regretted that specific results cannot be presented in this paper due to the conceptual nature of the work.

Hopefully, future reports can be more complete, and include not only detail stresses, but strengths of the graphite as well, the material development of which is being concurrently performed.

STRUCTURAL ANALYSIS — SPACE

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ANISOTROPIC ELASTIC-PLASTIC STRESS
ANALYSIS OF AXISYMMETRIC SOLIDS

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ABSTRACT

Advanced stress analyses for solid bodies of revolution are described and discussed. Emphasis is placed on the analysis of anisotropic materials that have temperature dependent properties and are exposed to very high temperatures, such as graphite in reentry vehicle nose tips.

Classical theories and various mathematical descriptions used in anisotropic elastic-plastic-thermal analyses are reviewed and compared. The constitutive equations presently being used and the OASIS code are described briefly, and it is shown that for application to graphite at reentry temperatures, the mathematical descriptions must be properly modified to account for anisotropy and temperature dependence effects.

The necessity for using both an incremental theory and an incremental method of solution to obtain an adequate description of what actually happens to the material is emphasized. The great difficulty of obtaining correct solutions without adequate material characterization and the requirement for optimum coding techniques to minimize computer time are also discussed.

INTRODUCTION

Advanced stress analyses for solid bodies of revolution via the finite-element method of solution are presented in this paper. Special attention is given to application of these analyses to anisotropic materials with temperature dependent properties, such as graphite for reentry vehicle nose tips. Features of the OASIS code are presented as appropriate throughout the paper.

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The first section reviews classical anisotropic thermal-plasticity theory. Various mathematical descriptions of a three-dimensional yield surface, the associated plastic flow law, and the appropriate inclusion of the temperature dependence are presented. The particular form of the incremental constitutive equations presently being used is also given.

The second section describes the finite-element method of solution, the types of elements used, the discreteness required, and the various methods of solving nonlinear problems, i.e., iterative vs incremental techniques.

The third section presents several numerical examples of both classical isotropic plasticity problems, to verify the method of solution, and hypothetical orthotropic thermal plasticity problems, to illustrate the importance of a correct orthotropic description. The fourth section demonstrates the necessity for the proper inclusion of temperature rate effects.

The fifth section describes work being performed on graphite reentry vehicle nose tips. Topics concerning the temperature dependence of graphite material properties, tension-compression differences for both elastic and plastic regimes, yield surfaces and plastic volume changes, and appropriate constitutive equations are discussed. The results of several sample problems of graphite materials loaded by both mechanical and thermal loads are given. In addition, comparisons for solving these problems by both iterative and incremental methods of solution are presented.

The concluding remarks summarize some of the most critical aspects of elastic-plastic-thermal analyses, including optimum coding techniques and economics of obtaining accurate solutions for nonlinear problems such as anisotropic thermal plasticity solutions.

INCREMENTAL ANISOTROPIC ELASTIC-PLASTIC CONSTITUTIVE EQUATIONS

The complete theory describing the development of the anisotropic, elastic-plastic-thermal, incremental constitutive equations used in the OASIS finite element computer code, together with the constant-strain finite element derivation, is given in Ref. 1. The anisotropic plasticity

theory is based upon the classical concepts of a yield condition and its associated flow law. The anisotropic yield condition is an extension of the work of Jensen, Falby, and Prince.² The associated flow law is derived for the non-isothermal state by applying a set of restrictive conditions originally given by Prager.³ The essential results of the derivation consist of the following:

- o Derivation of the anisotropic parameters for bilinear stress-strain curves, following Ref. 2.
- o Derivation of the plasticity matrix and plasticity vector through use of Prager's restrictive conditions (Ref. 3).
- o Derivation of incremental equations used in the tangent stiffness method, following the development presented in Refs. 4, 5, and 6.

The results of these derivations are given in the following.

First, define the vectors and matrices:

$$\{\sigma\} = \begin{Bmatrix} \sigma_r \\ \sigma_z \\ \sigma_\theta \\ \sigma_{rz} \end{Bmatrix} \quad \{\epsilon\} = \begin{Bmatrix} \epsilon_r \\ \epsilon_z \\ \epsilon_\theta \\ \gamma_{rz} \end{Bmatrix} \quad \{\alpha\} = \begin{Bmatrix} \alpha_r \\ \alpha_z \\ \alpha_\theta \\ 0 \end{Bmatrix}$$

$$[\alpha] = \begin{bmatrix} \alpha_{rr} & \alpha_{rz} & \alpha_{r\theta} & 0 \\ \alpha_{rz} & \alpha_{zz} & \alpha_{z\theta} & 0 \\ \alpha_{r\theta} & \alpha_{z\theta} & \alpha_{\theta\theta} & 0 \\ 0 & 0 & 0 & \beta_{rz} \end{bmatrix}$$

Notice that the anisotropic parameter matrix $[\alpha]$ is to be distinguished from the thermal expansion vector $\{\alpha\}$. In addition, the components of all stress vectors and strain vectors are ordered as shown by $\{\sigma\}$ and $\{\epsilon\}$ above.

The equivalent stress $\bar{\sigma}$ is defined by

$$\bar{\sigma}^2 = \{\sigma\}^T [\alpha] \{\sigma\}$$

where the anisotropic parameters α_{ij} are given for a material whose principal stress-strain diagrams may be represented in bilinear form by the following expressions:

$$\alpha_{rr} = \left(\xi_r + \frac{\eta_r}{\sigma^2} \right)^{-1}$$

$$\alpha_{zz} = 1.0$$

$$\alpha_{\theta\theta} = \left(\xi_\theta + \frac{\eta_\theta}{\sigma^2} \right)^{-1}$$

$$\beta_{rz} = \left(\xi_{rz} + \frac{\eta_{rz}}{\sigma^2} \right)^{-1}$$

$$\alpha_{rz} = \frac{1}{2} (\alpha_{zz} + \alpha_{rr} - \alpha_{\theta\theta})$$

$$\alpha_{r\theta} = \frac{1}{2} (\alpha_{rr} + \alpha_{\theta\theta} - \alpha_{zz})$$

$$\alpha_{z\theta} = \frac{1}{2} (\alpha_{\theta\theta} + \alpha_{zz} - \alpha_{rr})$$

and

$$\xi_r = \frac{K_r}{K_z}, \quad \eta_r = Y_r^2 - \xi_r Y_z^2$$

$$\xi_\theta = \frac{K_\theta}{K_z}, \quad \eta_\theta = Y_\theta^2 - \xi_\theta Y_z^2$$

$$\xi_{rz} = \frac{K_{rz}}{K_z}, \quad \eta_{rz} = k_{rz}^2 - \xi_{rz} Y_z^2$$

$$\frac{1}{K_i} = \frac{1}{(Et)_i} - \frac{1}{E_i}, \quad i = r, \theta, z, rz$$

where E = Young's moduli, E_t = tangent moduli, Y = tension yield stress, and k = shear yield stress.

The z-direction stress-strain diagram was arbitrarily chosen as the equivalent stress-equivalent strain diagram, as discussed in Ref. 2. Consequently, $\xi_z = 1$ and $\eta_z = 0$, making $\alpha_{zz} = 1.0$. Defining

$$\frac{\partial(\quad)}{\partial \bar{\sigma}} = (\quad)', \quad \frac{\partial(\quad)}{\partial T} = (\quad)'$$

$$\Lambda = 1 - \frac{1}{2\bar{\sigma}} \{\sigma\}^T [\alpha'] \{\sigma\}$$

$$\{S\} = [\alpha] \{\sigma\}$$

$$\dot{\bar{\sigma}}^2 = \{\sigma\}^T [\dot{\alpha}] \{\sigma\}$$

then the "plasticity matrix $[D^P]$ " and the "plasticity vector $\{\phi\}$ " are given by

$$[D^P] = \frac{1}{K\bar{\sigma}^2 \Lambda} \{S\} \{S\}^T$$

$$\{\phi\} = \frac{1}{K\bar{\sigma}^2} \left[\frac{\dot{\bar{\sigma}}^2}{2\Lambda} - \frac{1}{2} \left(\bar{Y}^2 - \bar{Y}_0^2 \right) \frac{\dot{K}}{K} - \bar{Y}_0 \dot{\bar{Y}}_0 \right] \{S\}$$

where $\bar{Y}$ is the equivalent yield stress whose initial value is $\bar{Y}_0$. The value of $\bar{Y}$ increases from its initial value $\bar{Y}_0$ due to work hardening, as expressed by the tangent modulus E_t . In addition, K = the plasticity modulus defined above.

Finally, the incremental plastic strain $\{d\epsilon^P\}$ is related to the incremental stress $\{d\sigma\}$ and the incremental temperature dT by

$$\{d\epsilon^P\} = [D^P] \{d\sigma\} + \{\phi\} dT$$

In a similar manner, the incremental elastic strain $\{d\epsilon^E\}$ is related to the incremental stress $\{d\sigma\}$ and incremental temperature dT by

$$\{d\epsilon^E\} = [D^E] \{d\sigma\} + \{\bar{\alpha}\} dT$$

where

$$\{\bar{\alpha}\} = \{\alpha\} + [\dot{D}^E]\{\sigma\} + \{\dot{\alpha}\}(T - T_0)$$

$[D^E]$ is the "elasticity matrix," and $\{\bar{\alpha}\}$ is the "thermal elasticity vector," with $\{\alpha\}$ being defined above and T_0 being the reference temperature.

Combining the elastic and plastic incremental strains to form the total incremental strain gives

$$\{d\epsilon\} = ([D^E] + [D^P])\{d\sigma\} + (\{\bar{\alpha}\} + \{\phi\})dT$$

Solving for $\{d\sigma\}$ yields

$$\{d\sigma\} = [C^{EP}]\{d\epsilon\} - \{d\tau\}$$

where the variable elastic-plastic coefficient matrix $[C^{EP}]$ is given by

$$[C^{EP}] = ([D^E] + [D^P])^{-1}$$

and the incremental thermal stress vector $\{d\tau\}$ is given by

$$\{d\tau\} = [C^{EP}](\{\bar{\alpha}\} + \{\phi\})dT$$

This method of solution is sometimes referred to as the variable stiffness method or tangent stiffness method (cf. Refs. 5, 6).

Combining the above incremental relations with any standard finite element representation for bodies of revolution eventually leads to a set of incremental, simultaneous, linear algebraic equations of the form

$$\{dF\} = [\bar{K}]\{du\}$$

where $\{dF\}$, the incremental force vector contains both $\{d\sigma\}$ and $\{d\tau\}$, $[\bar{K}]$ is the variable stiffness matrix, and $\{du\}$ is the incremental displacement vector. Since $[\bar{K}]$ is a function of the current stress and accumulated plastic strain, a separate problem must be solved for each increment of load. The resulting values of incremental stress and strain are added to the previous total values to form the new total values of stress and strain.

The above incremental constitutive equations were incorporated into the OASIS finite element code, which was developed to solve plasticity problems for bodies of revolution. A comprehensive description of the formulation is given in Ref. 1. The solution procedure is presented in flow chart form, and is given in Fig. 1. The incremental solution is found following a technique used by Yamada, Yoshimura, and Sakurai.⁷ The size of the incremental step is iteratively determined, such that the most highly stressed elastic element which is actively loading (increasing in stress level) just reaches a plastic state. The iterative procedure is required since both the incremental thermal loads and the stiffnesses of the elements are functions of the instantaneous temperature loads. In addition to this iterative procedure, there is a provision to limit the incremental step size to some predetermined fraction of the total load size. Additional features of the OASIS code are presented later.

PLANE STRAIN ELASTIC-PLASTIC SOLUTION OF AN ISOTROPIC HOLLOW CYLINDER LOADED BY INTERNAL PRESSURE

To be able to check the accuracy of elastic-plastic solutions obtained by the finite element code OASIS, it is necessary to obtain an independent solution of known validity. One of several axisymmetric elastic-plastic solutions available in the literature is an isotropic plane strain hollow cylinder loaded by internal pressure. Solutions to this problem have been obtained by both numerical^{8,9} and analytical¹⁰ methods for elastic-perfectly plastic materials (no hardening). However, a solution for a work hardening (or strain hardening) material does not appear to be available. Consequently, this problem was solved for a work (strain) hardening material whose stress-strain curve is expressed in bilinear form (to be published, cf. Ref. 11). The solution technique employed was to reduce the problem to a set of hyperbolic partial differential equations and obtain the solution by the method of characteristics, following the same general method presented in Ref. 9. The problem was also solved by the finite element method using the OASIS code. The finite element model consisted of 20 elements extending from the inside cylindrical radius $r = a$ to the outside radius $r = b$. This is

shown graphically in the bottom of Fig. 4. The radius of the elastic-plastic interface is designated $r = c$. For a plane strain solution, only one row of radial elements was needed to model the cylinder. The solution is for a strain hardening material which is represented in bilinear form (cf. insert, Fig. 4). Other geometric and material parameters are given as

$$b/a = 2.0, \quad E/k_0 = 1.0, \quad \nu = 0.30$$

$$H/E = 0.12162, \quad \alpha = 12.5 \times 10^{-6}, \quad Y/k_0 = \sqrt{3}$$

where E = Young's modulus, ν = Poisson's ratio, H = tangent modulus, α = coefficient of linear thermal expansion, Y = yield stress in pure tension, and k_0 = yield stress in pure shear. The bilinear stress-strain curve of this material is shown in the insert in Fig. 4. The results for this problem are shown in Figs. 2 to 5. Results obtained by the method of Ref. 11 are shown by solid lines, while results obtained by the finite element method are shown by points within the small circles. Figs. 2 through 4 show the non-dimensional stresses σ_r/k_0 , σ_θ/k_0 , and σ_z/k_0 ($\tau_{rz}/k_0 \equiv 0$ due to symmetry) for various stages of plastic flow. In Fig. 2, plastic flow is just ready to commence ($c/a = 1.0$), and hence this represents an elastic solution. In Fig. 3, the elastic-plastic boundary is half way through the cylinder, i.e., $c/a = 1.5$. Figure 4 shows the residual stresses when the applied internal pressure is reduced to zero from the state shown in Fig. 3. The relationship between the non-dimensional applied pressure (p/k_0) and the location of the elastic-plastic boundary (c/a) is shown in Fig. 5. The data points shown in Fig. 5 also indicate the incremental steps used in the finite element solution. A total of 11 steps (not including the initial elastic solution) were used to obtain the solution for $c/a = 1.5$. For all of the data shown in Figs. 2 to 5, the maximum percentage error (i.e., regarding the solution by the method of Ref. 11 as exact) was less than 0.2 percent.

It should be mentioned that due to the nature of the finite element method of solution (i.e., the displacement method), the stresses are the least accurate of all of the quantities found. The displacements are the most accurate, since these are the primary explicit unknowns in the

Rayleigh-Ritz type of solution procedure employed. The strains are found by calculating the rate of change of the displacements. Since the displacements are only known at discrete points (nodes), this rate of change must be approximated by a finite difference formula (which becomes exact as the nodes approach each other in the limit). Consequently, the strains are less accurate than the displacements. The stresses are related to the strains by the constitutive equations. When plasticity is present, these constitutive equations are functions of the total accumulated stresses (or strains). Since a finite increment size must be taken in any numerical solution, errors develop in the incremental strains and these errors accumulate in the total strains. Consequently, the constitutive equations contain errors and therefore the incremental stresses are less accurate than the incremental strains. Since both total stresses and strains are found by adding the incremental values, the total stresses are less accurate than the total strains.

By the reasoning presented above, the most severe test of the method is to examine the accuracy of the stresses. For the test problem presented here, the maximum error is less than 0.2 percent, which is extremely accurate.

COMPARISON OF INCREMENTAL THEORY AND DEFORMATION THEORY METHODS OF SOLUTION

In order to evaluate the accuracy of the OASIS incremental code in the case of materials with orthotropic properties, an independent solution, either numerical or analytical, was sought. A literature search failed to reveal any available inelastic solutions for orthotropic materials. Therefore, it was decided to seek comparisons between incremental solutions and deformation plasticity solutions* for cases in which the deformation theory could be expected to provide a good approximation to the incremental result.

An anisotropic elastic-plastic finite element computer code (DOASIS) based on deformation theory of plasticity was developed to make solutions

*Deformation plasticity theory neglects load path dependence of inelastic material behavior.

available for comparison with the incremental code (OASIS). The basic anisotropic plasticity theory used in the incremental code was used to generate the appropriate equations for the deformation theory code. The essential difference is that an incremental calculational procedure is not employed in the deformation theory code. Both codes were then used to solve problems of plane strain hollow cylinders composed of orthotropic materials and loaded by an assumed temperature distribution and by internal pressure.

For the temperature loading problems, the material properties are taken to be independent of temperature, and orthotropy is introduced by assuming that the yield stress in the hoop direction is different from the value for the radial and axial directions. Thus, the material has a plane of isotropy, the radial/axial plane. The material properties in the isotropic plane are given by:

$$E/Y = 208.0, \quad Y/k = \sqrt{3}, \quad \nu = 0.30, \quad \alpha = 12.5 \times 10^{-6}$$

In the orthotropic plane, $Y_\theta = \eta Y$, $E_\theta = E$, $\alpha_\theta = \alpha$, $\nu_{r\theta} = \nu_{z\theta} = \nu$, where E = Young's modulus, Y = yield stress, α = coefficient of linear thermal expansion, ν = Poisson's ratio, and η = some ratio factor. The assumed temperature distribution is given by

$$T(r) = \frac{T_0 a}{b - a} \left(\frac{b}{r} - 1 \right) \quad a \leq r \leq b$$

where T_0 is the temperature at the inside radius, $r = a$. The finite element model used by both codes consisted of 10 elements, extending from the inside radius $r = a$ to the outside radius $r = b$.

The incremental solution was obtained by first finding the value of T_0 which would just cause plastic flow to occur. The cylinder was then loaded with additional temperature increments $\Delta T(r)$, given by the above equation with ΔT_0 replacing T_0 . This was continued until the maximum temperature $T_0 = 800^\circ\text{F}$.

The deformation theory solution was obtained by applying the total temperature distribution, and iterating the solution until a preset convergence criterion was satisfied.

The non-dimensional stresses σ_r/Y , σ_θ/Y , and σ_z/Y comprising the elastic-plastic solution for the orthotropic plane strain hollow cylinder loaded by the above temperature distribution are given in Figs. 6 and 7 for two different degrees of orthotropy, namely

$$(a) \quad Y_\theta = 1.0Y \quad (\text{cf. Fig. 6})$$

$$(b) \quad Y_\theta = 0.5Y \quad (\text{cf. Fig. 7})$$

The solutions obtained by the incremental theory finite element code (OASIS) are shown by solid lines, and the solutions obtained by the deformation theory finite element code (DOASIS) are shown by circles (enclosing data points). In addition, the elastic solution, which is independent of the yield stresses and therefore applies to both solutions, is shown by a dashed line in Fig. 6.

In general, the incremental theory and deformation theory solutions agree quite well, with differences (where they did occur) being evident mainly in the regions of maximum plastic flow. The main reason for any differences to be present at all is that deformation plasticity theory does not account for path dependent effects arising from non-proportional loading* or unloading. However, for the two problems presented here, loading of each element was monotonically increasing and almost proportional, and consequently there were no significant path dependence effects. In addition, the material properties were assumed to be independent of temperature and, consequently, any path dependence that might be introduced by temperature dependent material behavior was not present. Hence, it was to be expected that the incremental and deformation plasticity theories would give essentially the same results for the inelastic response of the thermally loaded, hollow orthotropic cylinders. That the finite element computer codes corresponding to each of these formulations did in fact produce essentially the same orthotropic, elastic-plastic solution is a further indication of the validity of the formulation and coding of the OASIS computer program.

*It has been shown¹² that when all stresses increase proportionately, deformation plasticity theory gives results identical to incremental theory if conditions are isothermal or properties are not temperature dependent.

The above solutions provide an important illustration of the effects of inelastic orthotropy in materials. When the hoop yield stress $Y_\theta = Y$, plasticity initiated on the inside surface of the cylinder $r = a$ and spread outward under the specified thermal loading. This is shown in Fig. 6. Conversely, when $Y_\theta = \frac{1}{2} Y$, plasticity initiated on the outside surface of the cylinder $r = b$ and spread inward. This is shown in Fig. 7. Consequently, the elastic-plastic state of the plane strain hollow orthotropic cylinder with a specified loading is strongly dependent on the degree of orthotropy in the hoop direction. It is seen that the character of the orthotropic inelastic behavior can be quite different from that of an isotropic material under similar conditions.

ASSESSMENT OF CERTAIN EFFECTS ARISING FROM TEMPERATURE DEPENDENCE OF MATERIAL PROPERTIES

Temperature dependence of material properties gives rise to the presence of certain additional terms in the incremental constitutive relations. These terms involve the rate of change of material properties with respect to temperature. The influence of these terms is examined by solving an orthotropic cylinder problem. Two solutions are obtained: in one the terms being investigated are retained; in the other they are neglected.

The incremental, anisotropic elastic-plastic-thermal constitutive equations, given above, may be restated here in the following form:

$$\{d\epsilon^P\} = [D^P]\{d\sigma\} + \{\phi\} dT$$

$$\{d\epsilon^E\} = [D^E]\{d\sigma\} + \{\alpha\} dT + \left(\{\dot{\alpha}\}(T - T_0) + [D^E]\{\sigma\} \right) dT$$

Combining the elastic and plastic incremental strains yields

$$\{d\epsilon\} = ([D^E] + [D^P])\{d\sigma\} + \{\alpha\} dT$$

$$+ (\{\phi\} + \{\dot{\alpha}\}(T - T_0) + [D^E]\{\sigma\}) dT$$

The incremental strain due to a temperature increment dT has been divided into two parts. The first part is simply the elastic thermal strain in

incremental form. The second part contains three terms, all of which are attributable to temperature dependence of the material properties; i.e., these terms represent incremental strain due to the "rate of change" of the material properties with respect to temperature. They will be referred to herein as "temperature-rate" terms. To ascertain the effect that these temperature-rate terms have on elastic-plastic-thermal deformations, a plane strain hollow cylinder problem was solved both including and neglecting the influence of these terms. The material properties are taken to be orthotropic and temperature dependent. It is assumed that all of the material properties (with the exception of Poisson's ratio which is assumed constant) increase in value by 10 percent for every 100°F temperature rise. This may be expressed by the following relation; $f(T) = f_0(1 + 0.001T)$, where f represents a material property. The material properties are isotropic, with the following exception: orthotropy is introduced by assuming that the hoop yield stress $Y_\theta = 1.3Y$ (where Y is the isotropic value). The properties at zero temperature are given by

$$\frac{E}{k_0} = 1000.0 \quad \nu = 0.30 \quad \frac{H}{E} = 0.12162$$

$$Y_r = Y_z = Y \quad Y_\theta = 1.3Y \quad k_{rz} = \sqrt{3}Y$$

$$\alpha = 12.5 \times 10^{-6}$$

The temperature loading is taken as the steady state distribution

$$T(r) = T_0 \frac{\ln(r/a)}{\ln(b/a)} \quad a \leq r \leq b$$

where $T_0 = 250^\circ\text{F}$ is taken to be the steady state temperature of the outside surface $r = b$; the steady state temperature of the inside surface $r = a$ is zero, which is also the reference temperature. The finite element model consists of 20 elements, extending from $r = a$ to $r = b$.

The problem was solved with the OASIS code using two different methods. In the first method, all of the effects of material property variation with respect to temperature were retained. The results are shown by solid lines in Figs. 8 and 9. The second method of solution

neglected all of the "temperature-rate" terms but retained all other effects of property temperature dependence. These latter results are shown by dashed lines in Figs. 8 and 9. The nondimensional stresses σ_i/k_0 , $i = r, \theta, z$ (where k_0 is the shear yield stress at zero temperature) are given in Fig. 8. The nondimensional elastic strains $E_0 \epsilon_i^E/k_0$ and plastic strains $E_0 \epsilon_i^P/k_0$, $i = r, \theta, z$ (where E_0 is Young's modulus at zero temperature) are given in Fig. 9. The maximum differences occur at the outside surface $r = b$. The stresses are 11 percent to 13 percent low and the strains are 14 percent to 16 percent low when the temperature-rate terms are neglected. This error is, of course, dependent upon how the material properties vary with temperature. Yielding for both solutions first occurred for an outside surface temperature $T_0 = 120.7^\circ\text{F}$. The final surface temperature was $T_0 = 250.0^\circ\text{F}$. Hence, the temperature difference of 129.3°F produced a 12.93 percent change in material properties (at the outside surface $r = b$) from initiation of plastic flow to the final elastic-plastic state. For this particular problem, the error introduced by neglecting the above rate terms is of the same magnitude as the material property temperature dependence (i.e., the change in material properties corresponding to the temperature change for the problem).

This example has clearly shown that for materials with appreciably temperature dependent material properties, the correct elastic-plastic-thermal incremental method of solution must include the temperature-rate terms.

GRAPHITE INVESTIGATIONS

Results of graphite investigations are given in the following sections. In all cases to be discussed, graphite material property data were used, either actual data or bilinear representations (see Figs. 10 through 13 or Table 1). The method of obtaining the best fit "least-squares" bilinear representations of the actual stress-strain curves is explained in Ref. 1. The important aspect to consider is that equally spaced data points (along the strain axis) must be used to prevent biasing the results.

Incremental and Deformation Theory Comparison

A problem was selected to illustrate the differences between solutions obtained by deformation and incremental plasticity theories. A hollow cylinder loaded by the temperature distribution for $\tau = 0.10$, shown in Fig. 14, was investigated. The bilinear material property data of Table 1 were used. The elastic-plastic-thermal solution to this problem was obtained by both the incremental theory code (OASIS) and the deformation theory code (DOASIS).

The incremental solution was obtained as follows. A maximum load step size of 4 percent of the total thermal load was enforced during the solution. Since the properties (and therefore the stiffness matrix) are temperature dependent, the solution for each increment was calculated iteratively. This was necessary because the load increment* is not selected a priori, but is determined in the course of the solution procedure. Hence, the appropriate material property values are not known prior to calculating the solution for a given load increment, but must be estimated from previous results. A maximum of two iterations per increment was used. Seventeen load increments requiring a total of 13.02 seconds (central processor time) on a CDC 6600 computer were used in obtaining the incremental solution.

For the deformation plasticity theory solution the total load is applied in a single increment, but the calculation of the inelastic response requires iterative computational procedures. A new estimate of the total plastic strain in each finite element is calculated in each iteration. The properties, and therefore the stiffness matrix, are functions of the total temperatures in this type of solution. Calculated results from each iteration are compared with results of the previous iteration, and the calculation is terminated on the basis of a preset convergence criterion. A total of 10 iterations were required by the DOASIS code to obtain the elastic-plastic-thermal solution within an error limit of 0.1 percent for the effective stresses.

*The load increment includes the temperature change.

The stress and strain components obtained by the above two solutions are shown in Figs. 15 and 16. The dashed curves are the incremental (OASIS code) results and the solid curves the deformation theory (DOASIS code) results. Significant differences exist between these two solutions:

- o In the incremental solution, the final state is elastic from $r/a = 1.0$ to $r/a = 1.65$, plastic from $r/a = 1.65$ to $r/a = 1.85$, and elastically unloaded from $r/a = 1.85$ to $r/a = 2.00$. To indicate this unloading from a plastic state, the maximum stress level reached during the incremental solution is also shown in Fig. 15 for elements in the region $r/a = 1.85$ to 2.00 . This maximum stress envelope is indicated only for the axial (σ_z) component of stress.
- o In the deformation solution, the final state is elastic from $r/a = 1.00$ to $r/a = 1.65$ and plastic from $r/a = 1.65$ to $r/a = 2.00$.
- o The predicted elastic and plastic strains are completely different in the inelastic region. Since unloading occurred from $r/a = 1.85$ to $r/a = 2.00$ in the incremental solution, only small plastic strains were developed. Conversely, large plastic strains were predicted in the deformation theory solution.
- o The total maximum equivalent strains based upon the distortion energy theory, i.e., $\bar{\epsilon} = [2/3 \{(\epsilon_r - \epsilon_\theta)^2 + (\epsilon_\theta - \epsilon_z)^2 + (\epsilon_z - \epsilon_r)^2\}]^{1/2}$ were completely different. The maximum equivalent strain in the deformation theory results occurred at $r/a = 2.0$ and had a value of $\bar{\epsilon}_{\max} = 0.02229$; the corresponding maximum equivalent strain predicted for $r/a = 2.0$ by the incremental theory was $\bar{\epsilon} = 0.00857$ (notice that $\bar{\epsilon}_{\max}$ did not occur at $r/a = 2.0$ for the incremental theory results). Hence, completely erroneous conclusions could be drawn from the deformation theory results for this problem if the failure criterion were based upon the maximum equivalent strain.
- o The stresses predicted by the two theories are in relatively good agreement, the maximum difference being less than 7 percent. Hence, the failure criterion based upon maximum equivalent

stress for this problem would produce better agreement between the two theories than one based upon maximum equivalent strain. Both of these solutions were obtained for a monotonically increasing thermal load. Hence, an important unloading phenomenon was observed. The unloading of a portion of the structure under monotonically increasing thermal load apparently resulted from the temperature dependence of the material mechanical properties.

Such path dependent phenomena cannot be predicted with a deformation plasticity code, because arbitrary load histories are not accounted for. In general, an incremental formulation is required to correctly and reliably predict detailed thermostructural response when load path dependent effects, such as temperature dependent inelastic behavior, are important. The deformation plasticity theory code is a useful tool for rapidly obtaining approximate answers for engineering design, but the validity of such computational results can only be assessed by an incremental analysis.

Sensitivity Analyses

Sensitivity studies were performed using the OASIS code to explore material parameter sensitivity of elastic-plastic-thermal solutions. Best-fit bilinear stress-strain representations for graphite were used. The results of these studies are described below.

The sensitivity study consisted of a parametric investigation of key material constants which affect the elastic-plastic solution. The two parameters which directly affect the plastic flow characteristics of orthotropic materials represented by bilinear stress-strain curves are the "tangent modulus" (or "strain hardening modulus") and the "yield stress." For orthotropic materials these quantities have independent values in each principal material direction. The elastic constants of the material affect the plastic flow only indirectly through mechanical interaction of elastic and plastic zones in the material.

The values of the tangent moduli (E_t) and yield stresses (Y) in the principal material directions were varied in both pressure loading and thermal loading cases to ascertain their effect on the elastic-plastic-

thermal solution of these types of problems. Four parametric variations were calculated for the pressure loading problem:

- o E_t was increased by 50 percent and 100 percent.
- o All yield stresses were increased by 20 percent.
- o The transverse yield stresses (Y_r and Y_θ) were increased by 20 percent.

Two parametric variations were calculated for the thermal loading problem:

- o E_t was increased by 100 percent.
- o All yield stresses were increased by 20 percent.

The geometries, material properties (bilinear characterization), and loadings were the same as in the previous comparison study of incremental and deformation theories. The sensitivity study results for the pressure loading problem are shown in Fig. 17 and the results for the thermal loading problem are shown in Fig. 18. Results from the pressure loading cases indicate several trends. First, for a 100 percent increase in E_t , the maximum change in stress level was approximately 12 percent, and the location of the elastic-plastic boundary changed by only 6 percent. For the 20 percent increase in yield stress, the maximum change in stress level was approximately 12 percent in the hoop stress component and 40 percent in the axial component, with the location of the elastic-plastic boundary changing by 23 percent. Thus, for this pressure loading problem, the results are relatively insensitive to variations in tangent modulus values, but variations in yield stress values produce effects which can be of the same order as the change in yield stress. Examination of the results of the thermal loading problems leads to similar conclusions.

CONCLUSIONS

In order to perform accurate stress analyses of structures composed of graphite materials, the inelastic, temperature dependent behavior of graphite must be included in the mathematical characterization. Since most graphites have different stress-strain responses under tensile and compressive loading conditions, this multi-modulus behavior must also be included. The theory presented in this paper is for a mono-modulus material, i.e., tensile and compressive responses are assumed the same,

and consequently only partially represents the actual behavior of graphites. If a new theory is developed similar to the one presented in this paper, it would have to have a multi-modulus, orthotropic yield surface description, along with an associated plastic flow law which includes the thermal plasticity terms described herein. For materials with significant temperature dependence, such as graphites, the incremental elastic-plastic-thermal constitutive equations must include the temperature dependent rate terms. The method of solution should be incremental rather than a total loading technique (deformation plasticity theory) to properly account for the non-proportional loading effects which occur under general loading conditions. This cannot be accomplished by a deformation plasticity theory method of solution, although such a theory is useful under certain conditions.

Optimum coding techniques are needed in any resulting computer program to make it an economical and useful tool. This is especially true for computer programs which solve plasticity problems via an incremental method of solution. The solution to each problem can involve many stages of incremental loading, and many iterations per increment. Hence, optimization of the coding will minimize the computer run times and make the computer program economically feasible for general use. The eventual use of an incremental plasticity computer program requires accurate, representative material property data, throughout the temperature range of interest. For space applications, this requires property data from room temperature to about 6500°F, since reentry vehicles are subject to these high temperatures. The type of material property data needed will involve both uniaxial and biaxial loading tests. This puts a severe requirement on experimentalists. In addition, theoreticians and experimentalists should work together to obtain the correct material description and resulting data to be used in the final analysis.

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Table 1. Bilinear Stress-Strain Data for Graphitite G-90 Graphite

Temperature (°F)	Axis ^a	Y (psi)	ϵ_Y ($\times 10^3$ in./in.)	E ($\times 10^{-6}$ psi)	E_t ($\times 10^{-6}$ psi)	ν
80	L	5388.328	5.026827	1.072991	0.1831567	0.135
	T	5593.458	7.752211	0.7208499	0.1661219	
2000	L	5311.787	3.725127	1.427323	0.1867635	0.135
	T	6665.154	11.34327	0.5880451	0.1191039	
3000	L	6901.462	4.981286	1.386835	0.08740177	0.135
	T	6772.054	14.07741	0.4806089	0.1267082	
4000	L	6806.717	7.247506	0.9401036	0.07885231	0.133
	T	5795.440	7.872204	0.7358049	0.1207527	
5000	L	2964.404	6.654199	0.4450626	0.07033704	0.130
	T	2964.404	6.654199	0.4450626	0.07033704	

^aL = longitudinal; T = transverse.

Fig. 2. Elastic Stresses in a Hollow, Isotropic, Plane Strain Cylinder Loaded by Internal Pressure ($c/a = 1.00$).

Fig. 3. Elastic-Plastic Stresses in a Hollow, Isotropic, Plane Strain Cylinder Loaded by Internal Pressure ($c/a = 1.50$).

Fig. 4. Residual Stresses in a Hollow, Isotropic, Plane Strain Cylinder Loaded by Internal Pressure (Unloaded from $c/a = 1.50$).

Fig. 5. Nondimensional Pressure (p/k_0) vs Elastic-Plastic Boundary Location (c/a) of a Hollow, Isotropic, Plane Strain Cylinder Loaded by Internal Pressure.

Fig. 6. Elastic-Plastic Stresses in a Hollow, Orthotropic ($Y_\theta = Y$) Plane Strain Cylinder Loaded by a Temperature Distribution.

Fig. 7. Elastic-Plastic Stresses in a Hollow, Orthotropic ($Y_\theta = 0.5Y$) Plane Strain Cylinder Loaded by a Temperature Distribution.

Fig. 8. Elastic-Plastic Stresses in a Hollow, Orthotropic ($Y_\theta = 1.3Y$) Plane Strain Cylinder Loaded by a Steady-State Temperature Distribution — Rate Dependence Study.

Fig. 9. Elastic and Plastic Strains in a Hollow, Orthotropic ($Y_\theta = 1.3Y$) Plane Strain Cylinder Loaded by a Steady-State Temperature Distribution.

Fig. 10. Longitudinal (With-Grain) Stress-Strain Diagrams of Graphitite G-90 Graphite at Temperature.

Fig. 11. Transverse (Across-Grain) Stress-Strain Diagrams of Graphitite G-90 Graphite at Temperature.

Fig. 12. Bilinear Material Property Data (Elastic and Tangent Moduli) vs Temperature.

Fig. 13. Bilinear Material Property Data (Yield Stresses) vs Temperature.

Fig. 14. Temperature Distributions in a Hollow Cylinder for Various Times.

Fig. 15. Elastic-Plastic Stresses in a Hollow, Plane Strain Graphite Cylinder Loaded by a Thermal Gradient - Incremental and Deformation Theory Comparisons.

Fig. 16. Elastic and Plastic Strains in a Hollow, Plane Strain Graphite Cylinder Loaded by a Thermal Gradient - Incremental and Deformation Theory Comparisons.

Fig. 17. Elastic-Plastic Stresses in a Hollow, Plane Strain Graphite Cylinder Loaded by External Pressure - Tangent Modulus and Yield Stress Sensitivity Study.

Fig. 18. Elastic-Plastic Stresses in a Hollow, Plane Strain Graphite Cylinder Loaded by a Temperature Distribution - Tangent Modulus and Yield Stress Sensitivity Study.

ASAAS - ASYMMETRIC STRESS ANALYSIS OF AXISYMMETRIC SOLIDS

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ABSTRACT

A description is presented of a finite element method and the associated computer program to solve the thermal stress problem of asymmetrically heated axially symmetric bodies with temperature-dependent properties. The program is presently oriented towards analysis of graphite nosetips on reentry vehicles that undergo angle-of-attack heating, but it can be applied with only slight modification to a wide class of three-dimensional stress analysis problems. At the present time, the analysis is restricted to elastic, orthotropic material properties.

A two-dimensional axisymmetric triangular element is extended to a three-dimensional representation of the media by a Fourier series expansion of the problem variables in the circumferential direction. In the case of material properties that do not vary circumferentially, the system of resulting equations uncouples into the individual harmonics and solutions for each harmonic can be obtained independently. In the present case, the material properties are allowed to have circumferential variations, the harmonics are fully coupled, and the solutions of all the equations must be obtained simultaneously.

By ordering the simultaneous equations in a particular manner, a banded stiffness matrix is formulated. The solution is obtained through the use of a banded square-root Cholesky scheme which is blocked according to the amount of immediate-access storage available in the particular computer being used. For a given amount of computer storage, there is a limit on the number of harmonics and nodal point connectivity that can be employed. However, with the present generation of computers, ample storage is available for solving most practical problems.

INTRODUCTION

The difficulty of performing stress analyses of graphite structures has been described in many papers, including a number presented at this conference. The reasons that it is necessary to account for plasticity, anisotropy, viscoelasticity, nonlinear effects, time effects, stochastic processes, etc., have been discussed. These and other factors make graphite stress analysis very difficult.

This paper adds one more element of complexity to the general problem, at the level of material behavior that can be described by the theory of elasticity; this added complexity is the extension of stress analyses to three-dimensional problems. In common with many methods of analysis, the method presented here is applied with the assumption of linear elasticity but can later be applied to problems of plasticity, viscoelasticity, etc.

There is an interesting class of reentry problems being investigated at present that requires three-dimensional analysis methods: these include graphite nosetips that are subjected to asymmetric heating, such as would occur during angle-of-attack flight. This presentation describes a three-dimensional method of analysis designed to solve for thermal stresses in graphite nosetips subjected to angle-of-attack heating. Of course, the method is not restricted to that particular problem.

The product of this research is a computer program, ASAAS, an acronym for Asymmetric Stress Analysis of Axisymmetric Solids. It has been written, debugged, and applied to some relatively simple problems. This presentation is only a status report in the sense that there remains considerable development work for optimizing run times, improvement of accuracy, etc.

Figure 1 illustrates the general mechanical problem being solved. Note the R-Z- θ polar coordinate system. This is an arbitrary axisymmetric solid which, in general, may consist of an elastic, orthotropic material with temperature-dependent properties. A unique feature of this solution method is that the material properties can vary in the circumferential direction, which, of course, is important for graphite thermal stress analysis in problems such as angle-of-attack heating because of the temperature dependence of the properties.

A typical idealization of the solid is shown in Fig. 2. In two dimensions the solid is discretized by a set of arbitrary quadrilateral elements. Each quadrilateral element is divided into four triangles. The triangular element is the basic element of the computer program but the subdivision into triangles is accomplished internally, so that to the stress analyst the modeling element is the quadrilateral.

BASIC EQUATIONS

The basic equations that characterize the method of analysis are used for a Fourier series expansion of the displacements (which are dependent variables), linearization of each Fourier coefficient for the displacement function in a triangular element, variation of material properties, application of the theory of minimum potential energy, and coupled theta integrations. These equations are displayed and discussed below.

Fourier Series Expansion of Displacements

The third-dimension modeling is accomplished by a Fourier series representation of all of the dependent variables. The first three equations show the expansion of the displacement field into a Fourier series form:

$$u_r(r, z, \theta) = \sum_{m=0}^M u_{rm}(r, z) \cos m\theta$$

$$u_z(r, z, \theta) = \sum_{m=0}^M u_{zm}(r, z) \cos m\theta$$

$$u_\theta(r, z, \theta) = \sum_{m=0}^M u_{\theta m}(r, z) \sin m\theta$$

Since the finite element method of solution is employed herein, the displacements are the unknowns of the problem. An implicit assumption of the method is that M , the total number of harmonics required for a solution, is not greater than about 6. If it is necessary to account for more harmonics, a general three-dimensional finite-element analysis would probably be more efficient.

Linear Displacement Field

Each Fourier coefficient for the displacement function in a triangular element is linearized in the standard way:

$$u_{rm} = A_{1m} + A_{2m}r + A_{3m}z$$

$$u_{zm} = A_{4m} + A_{5m}r + A_{6m}z$$

$$u_{\theta m} = A_{7m} + A_{8m}r + A_{9m}z$$

This results in $9M$ generalized coordinates that represent the displacement field in a triangular element, where M is the number of harmonics discussed above.

Material Properties

The equation for material properties is

$$\begin{bmatrix} c \end{bmatrix}_{6 \times 6} = \sum_{\bar{m}=0}^{\bar{M}} \begin{bmatrix} c_{\bar{m}} \end{bmatrix}_{6 \times 6} \cos \bar{m}\theta$$

As mentioned in the Introduction, a unique feature of this method is that the material properties can vary in the circumferential direction; this is done by expanding the stress-strain matrix into a Fourier series. The Fourier coefficients for the material terms, however, are automatically

generated within the program, and they arise from the temperature dependence of the properties.

Theory of Minimum Potential Energy

The problem is formulated by applying the theory of minimum potential energy:

$$\delta V = 0$$

where the total potential energy is the difference between the strain energy and the work done by internal and external forces,

$$V = U - \int_{\text{vol}} u_i F_i dV - \int_{\text{area}} u_i P_i dA$$

By minimizing the total potential energy, a system of linear simultaneous equations is obtained, which can then be solved by standard methods.

Coupling of Theta Integrations

The complexity of the problem arises due to coupling of the theta integrations:

$$\sum_{\bar{m}=0}^{\bar{M}} \int_0^{2\pi} [C_{\bar{m}}] \cos m\theta \cos \bar{m}\theta \cos \tilde{m}\theta d\theta$$

and

$$\sum_{\bar{m}=0}^{\bar{M}} \int_0^{2\pi} [C_{\bar{m}}] \sin m\theta \cos \bar{m}\theta \sin \tilde{m}\theta d\theta$$

These integrations are necessary in the formulation of the stiffness matrix. In each of these integrations, the three trigonometric terms involving m , $\bar{m}$, and $\tilde{m}$ are derived, respectively, from force (load) considerations, material behavior, and displacement. Note that in formulating the stiffness terms, it is necessary to sum and integrate a triple product of cosine terms. It can be seen that if the material properties were constant in the circumferential direction, that would imply that $\bar{M}$ is equal to zero and the trigonometric term $\cos \bar{m}\theta$ would drop out. The equation would thus be reduced to a form in which the orthogonality condition applies. In that case the only non-zero combinations of integrations occur when the load and the displacement harmonics are the same ($m=\tilde{m}$), and the system of equations can be uncoupled into a problem that can be solved for one harmonic at a time. But such is not the case here: the material properties vary circumferentially, the harmonics are fully coupled, and all equations must be solved simultaneously.

CONVENTIONAL AND ASAAS MATRICES

The magnitude of the problem can be seen in Fig. 3, which illustrates the form of the complete body stiffness matrix. Since the harmonic analysis method has been applied previously to problems that uncouple, the natural extension to a coupled system gives rise to a stiffness matrix that can be represented by the conventional form on the left in Fig. 3. This diagram shows the relative locations and numbers of the non-zero terms. Each minor diagonal represented by a line is nine terms wide. The total size of the system is 3000 equations, which are necessary for solving a problem of 216 elements and four harmonics. If the problem uncouples, all of the off-diagonal submatrices are zero and each of the four diagonal submatrices is independent. In that case, the problem is simplified to the point that four independent solutions of 750 banded equations are all that is necessary. However, with full coupling, present-day computers cannot solve the problem efficiently in that form.

In the ASAAS method, by reordering the terms in the displacement vector and the load vector we achieve a banded form illustrated on the right in Fig. 3. This same problem in ASAAS has a stiffness matrix that

still represents 3000 equations but the matrix now has a semi bandwidth of only 144 terms. By carefully conserving core storage and blocking in a particular way, we can obtain efficient solutions with today's computers by a modified square-root Cholesky scheme.

APPLICATION TO TEST PROBLEMS

The first example problem is the stress analysis of isotropic infinitely long solid circular cylinders subjected to $\cos \theta$ and $\cos 2\theta$ pressure distributions. A closed-form plane strain solution of this problem may be obtained by the use of Ref. 1. Figure 4 shows the closed-form solution for displacements denoted by solid lines and the result of a four-element, two-harmonic solution by ASAAS denoted by open triangles.

The second example introduces the effects of thermal stress and material anisotropy in the form of an infinitely long hollow circular cylinder subjected to a radial and circumferential temperature variation. The pertinent parameters of the problem are shown in Fig. 5, along with a plane strain closed-form solution from Ref. 2 and two ASAAS solutions denoted by open circles and triangles. The triangles represent a four-element, two-harmonic solution, and the circles represent a ten-element solution. The fact that the ten-element idealization is closer to the closed form than the four-element solution serves to illustrate convergence of the method to the true answers with decreasing element size.

The unique feature of the program, the ability to accommodate temperature dependence and variation of material properties, is demonstrated by the problem illustrated in Figs. 6 and 7, with solutions in Figs. 8 and 9. In Fig. 6, a hollow cylinder with an applied temperature distribution is treated as a plane strain problem with the SAAS III computer program and is modeled with 72 quadrilateral elements. Note that in Figs. 6 and 7 for the plane strain problem, a rectangular coordinate system is used. The rectangular R-Z coordinates are in the same plane as the polar R- θ coordinates used generally throughout the paper but do not otherwise correspond with the polar coordinates. In ASAAS, the same problem was modeled with four elements in the R direction of the R-Z plane and three harmonics.

The isothermal lines in Fig. 7 show the temperature distribution that was input to the problem. This is similar to the temperature distribution in the shell portion of a nosetip subjected to angle-of-attack heating. The material properties used in both programs were temperature-dependent in a way similar to graphite.

As a point of reference, the problem was solved first by letting the material properties be constant; that is, not a function of temperature. Figure 8 shows the results obtained that way. This is a three-dimensional plot of the maximum principal stress versus the angle θ and the radial location. The lines are the results from ASAAS, and the circles are the results from SAAS III. Both methods are working the same problem, but in a different way, and the results agree excellently. With temperature dependence in the θ direction, the results achieved with both programs are shown in Fig. 9. Again the answers agree very well, so it is concluded that the temperature dependence is adequately accounted for in the new program.

SUMMARY

A method of analysis has been presented that can be used as a design tool in evaluating the thermal stresses in graphite nosetips subjected to angle-of-attack heating. The method has been demonstrated to be accurate by comparison of specialized sample problems to other available solutions. The associated computer program exists in breadboard form and is currently being refined for industry use. In the next few months, problems of accuracy will be studied and the program will be made user-oriented, documented, and applied to reentry problems. Eventually it will be made available to those who may have a use for it. When finished, the program should accommodate finite element idealizations of up to 250 elements and six harmonics on computers with core sizes of 60,000 words or more. It is estimated that run times will be in the range of 5 to 30 minutes on an IBM 360 computer, depending on the complexity of the problem.

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Fig. 1. Actual Solid.

Fig. 2. Finite Element Approximation.

Fig. 3. Location of Nonzero Terms in K .

Fig. 4. Harmonic Axisymmetric Plane Strain.

Fig. 5. Thermal Stresses in a Transversely Isotropic, Hollow Circular Cylinder.

Fig. 7. Temperature.

Fig. 6. Element Plot.

Fig. 8. Maximum Thermal Stress in Infinite Cylinder with Non-Temperature-Dependent Properties.

Fig. 9. Maximum Thermal Stress in Infinite Cylinder with Temperature-Dependent Properties.

X THERMAL/STRUCTURAL ANALYSIS OF DESIGNS
USING GRAPHITE MATERIALS

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ABSTRACT

The development of digital computer codes has made it possible for the analyst to accurately predict the behavior of large complex structures including shells, frames, and general three-dimensional solids. The finite element codes have been extensively developed for this purpose and are routinely used to analyze a variety of graphitic designs.

This paper presents the application of several of these codes to a range of structural problems and gives specific examples of designs using graphitic materials. Illustrative examples are shown of the computer plot routines that automatically obtain contour maps and data plots of stresses, strains, temperatures, etc., and thereby reduce the time consuming, error-prone, manual manipulation of the results.

The graphical input-output data routines of the computer codes enable the analyst to more easily and rapidly interpret the results and aid him in conveying this information to others. Of particular interest are the input data plots which eliminate errors and assure that the problem solved is indeed the one of interest.

The development of digital computer codes has enabled the engineer to solve problems that would have been impossible or impractical to solve a few years ago. Finite element codes have been extensively developed for the purpose of analyzing large complex structures and are routinely used to analyze a variety of graphitic designs.

When using graphitic materials, the most difficult problem encountered by the designer results from its extreme brittle nature. Special attention must be given to geometric details that would normally be unimportant in a structure made of a ductile material, where small errors in analysis usually result in insignificant local yielding. However, when using brittle graphites, such errors often lead to catastrophic failure. Therefore, successful use of graphite as a structural material requires analysis based on accurate geometric and material property data.

This paper discusses some aspects of the analysis techniques now being used by the structural engineer. The programs discussed have been highly developed for use in everyday structural analysis, and are used to rapidly solve critical design problems and to predict structural capability. They have automatic input-output routines that enable the engineer to easily interpret the results and to convey this information to others.

To aid the engineer in determining if the input data is error free, automatic mesh generators and graphic displays are used such as illustrated in the example in Figure 1. This particular example, of a juncture between a rocket motor case and the motor nozzle, illustrates the program's ability to combine different materials. The figure shows the overall geometry of the motor case and indicates a specific region, labeled as A-A, that is analyzed in detail. The analyst used the simplified coordinate to input the data, specifying the actual location of only a few selected points which are labeled A through J. This procedure enables him to rapidly describe to the computer a complex structure in the simplest terms. The computer maps the input data into the actual geometry of the structure and automatically determines the coordinates of all nodal points. From these displays the engineer can check the analytical model for errors and can judge whether he has satisfactorily described the structure.

After the problem has been adequately described to the computer, the required analysis can be performed. In Figures 2 and 3, typical output data plots illustrate the program automation used in analyzing a reentry vehicle nosetip. In this example, the temperature and eroded shape are determined by the thermodynamic program and are transferred to the structural program by means of magnetic tape. This automatic transfer of data saves the engineer from performing a very boring and tedious task and therefore eliminates many human errors.

Since the erosion, temperature, thermal gradients, and thermal strains are all strongly dependent on the initial geometry, many iterations of the thermal and structural analyses are required in order to obtain the best combination of geometric details. This type of iterative procedure can easily lead to long and drawn out design schedules or unsuccessful designs unless automated programs are used.

Figures 2 and 3 show the eroded shape, temperature and the resulting strains at various altitudes during reentry. These contour maps enable the engineer to quickly evaluate the overall performance of a particular design and aid him in locating the problem areas that require examination in greater detail. Additional automatically generated data plots enable the analyst to examine these areas without performing the time-consuming task of manually plotting the data. An example, shown in Figure 4, gives a typical plot of the temperature and thermal strain variation along the inside surface of a graphitic nosetip. These plots may, of course, be obtained for any output quantity such as stress, strain, temperature or deflection.

Although graphitic materials are brittle, they do exhibit some material nonlinearities, particularly in compression at high temperature. In order to make maximum use of graphite as a structural material, it is important to consider the effect of these nonlinearities in the analysis.

Structural codes have been developed that include the elastic-plastic nonlinear material effects. The usefulness of these codes is generally limited, however, because they often are applicable only to a very specific class of problem, they have prohibitive run times and, in many cases, lead to incorrect answers. To be an acceptable tool, these programs must have accurate incremental solutions, efficient computer run times and fast automatic input-output data displays.

Typical results from a program that meets these requirements are shown in Figures 5 and 6. This example is from an analysis of an aircraft disk brake which has been subjected to repetitive braking cycles and the corresponding changes in temperature. An examination of the results shows that, in this case, a great deal of plastic flow has taken place throughout the disk. Figure 5 gives the geometry of the disk and the analytical model used in thermal stress analysis. It also illustrates the regions in which plastic flow occurs and how these areas change during the cycle. Although the plots of plastic yielding are automatically generated by the computer, for clarity in this example, the finite element grid has been eliminated.

The stress-strain history of a particular point in the disk for the first two cycles is shown in Figure 6. The curve on the left gives the complete history for an element during the first cycle. It is apparent from this plot that as the brake heats up it yields and goes plastic; then, after a significant amount of yielding, decay in the thermal gradient results in an unloading of stress to the point where yielding occurs in the opposite direction. The curve on the right gives the continuation of the stress-strain history into the second cycle. Points labeled as A and B represent the residual stress at the end of the first and second cycles. This example, while producing significantly more plastic flow than would be apparent in the brittle graphites, illustrates the ability of this code

to account for the nonlinear material effects.

The analyst, however, has not been able to take full advantage of these finite element codes due to the limited structural material characterization that is available for graphite. The data that is available generally indicates extreme variations in the material properties from batch to batch and, as a result, the engineer has limited and many times worthless data to use for design. Too often he has been forced to make assumptions and approximations which may not be appropriate and, in many cases, must perform parametric studies in order to grossly predict the behavior of these designs. Success or failure with graphite has been very unpredictable and design has normally been obtained by trial and error. In order to significantly advance the use of this material valid failure criterion must be established, improvements in nondestructive and destructive tests for screening out defective billets must be developed, and variability of material properties between billets must be defined and minimized. Until these shortcomings are better defined and accounted for in the structural analysis, graphitic materials will continue to be misused more often than they are properly used.

VIEW A-A

GEOMETRY MAPPED
BY COMPUTER

CODE OF MATERIAL

1	FIBERGLASS	4	INSULATION
2	ALUMINUM	5	INSULATION
3	ELASTOMER	6	ELASTOMER

Fig. 1. Typical Finite Element Computer Model.

Fig. 2. Reentry Vehicle Nosetip Eroded Shape and Isotherms.

Fig. 3. Reentry Vehicle Nosetip Typical Strain Contour Maps.

Fig. 4. Reentry Vehicle Nosetip Typical Data Plots.

Fig. 5. Thermal Stress Analysis of an Aircraft Disk Brake.

STRESS-STRAIN HISTORY
FIRST THERMAL CYCLE

STRESS-STRAIN HISTORY
FIRST AND SECOND CYCLE

Fig. 6. Elastic-Plastic Thermal Stress Analysis of an Aircraft Disk Brake.

STRUCTURAL ANALYSIS OF CARBON AND
GRAPHITE FILAMENT WOUND HEATSHIELDS FOR
REENTRY HEATING ENVIRONMENT

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ABSTRACT

Structural analysis methods and results of a reentry-vehicle heatshield manufactured of carbon filaments are discussed. The matrix is deposited by carbon vapor deposition and subsequently heat treated at high temperatures. The analysis methods are an extension of orthotropic techniques used in analyzing glass-epoxy filament wound materials. Included is a basic description of some unique thermostructural problems, associated with filament wound carbon or graphite material, which do not occur in designing homogeneous, isotropic shells.

Summary

Structural analysis methods based on an extension of techniques used in analyzing glass-epoxy filament wound materials are presented for use in analyzing reentry-vehicle heatshields manufactured of carbon filaments with a carbon-vapor-deposited matrix. Results from an analysis of a heatshield made of CY2-5 rayon fibers and a heat-treated carbon-vapor-deposited matrix are included.

List of Symbols

- L - Direction of filament
- T - Direction perpendicular to filament
- σ_i - Stress for direction i, i = L, T and 1, 2
- T_{ij} - Shear stress
- ϵ_i - Strain for direction i
- γ_{ij} - Shear strain
- E_i - Orthotropic moduli of elasticity
- G_{ij} - Shear modulus of elasticity
- ν_{ij} - Poisson's ratios
- θ - Temperature rise
- α_i - Orthotropic coefficient of thermal expansion
- f - Superscript for freely oriented properties
- f - Subscript for fiber direction
- F_i - Allowable stresses for unidirectional lamina
- YL - Yield level
- N_i - Stress resultants
- M_i - Moments
- UTS - Ultimate tensile strength
- η_i - Distance from inside surface to surface i
- h - Total shell thickness
- ψ - Wrap angle
- δ - Fiber deflection

STRUCTURAL ANALYSIS OF CARBON AND
GRAPHITE FILAMENT WOUND HEATSHIELDS FOR
REENTRY HEATING ENVIRONMENT

Introduction

Recent use of filament wound "carbon-carbon" material for reentry-vehicle heatshields has required analysis of these filament wound shells subjected to thermal gradients through the shell thickness. In this paper, the techniques used in analyzing glass-epoxy filament wound materials are extended for use with carbon-carbon materials.

Macromechanic techniques are used to provide a bilinear model for the stress-strain behavior of the graphitized filament wound material. In the initial linear portion, the composite material is assumed to respond as a layered heterogeneous continuum. "Yielding" is determined by a modified Hill Anisotropic Failure Criterion. The second linear portion of the stress-strain curve is approximated by extending conventional netting analysis methods. The effects of residual stresses due to heat treatment at elevated temperatures, found to be very significant, are included. This material model is compared with data from room-temperature tests of filament wound cylinders in both uniaxial and biaxial stress states.

The material model is used to define the elevated-temperature characteristics of the composite, based on room-temperature test data. A second set of elevated-temperature stress-strain curves are presented which include estimates of temperature effects on the filaments and matrix material. Both sets of stress-strain curves are used to obtain heatshield thermal stresses due to axisymmetric shell heating. Response of the heatshield is approximated by considering a thin cylindrical shell with generalized plane strain conditions in the axial direction. Typical stress profiles and failure levels are discussed.

Bilinear Layered Material Model

The analysis of the filament wound (FW) carbon materials is based on the analysis of a layered shell composed of a series of orthotropic laminas. These laminas are orthotropic and are oriented at some angle to the shell axis, called the wrap angle. Normally the layers are oriented at both positive and negative wrap angles. The combination of two layers at positive and negative wrap angles will be called a balanced layup.

The stress-strain behavior of the graphitized FW material can be modeled as bilinear, as shown in Figure 1. The model can be described in four parts:

1. In the initial linear portion, the material responds like a heterogeneous continuum. The stress-strain properties for varying wrap angles can be calculated by using standard analysis methods such as References 1 and 2, where the basic assumptions are that each lamina is a homogeneous, orthotropic material and that strain compatibility exists at lamina faces.
2. The "apparent yield point" is determined primarily by matrix failure due to tension normal to the fibers and shear. This point is defined by using the modified Hill Failure Criterion, as presented in References 1 and 3.
3. The stiffness of the material in the second linear portion of the stress-strain curve is calculated by using some of the assumptions employed in conventional netting analysis.³ The stiffness is related to the lamina stiffness in the direction of the fibers and the wrap angle.

4. The ultimate strength also is related to the wrap angle and the strength in the direction of the fibers. This is calculated by using some assumptions from netting analysis. It should be noted that if this calculated ultimate strength is equal to or less than the "yield strength," the material will fail at the "yield stress." The stress-strain curve in this case will be linear to failure.

Figure 1. Material behavior

Elastic Behavior - Region One

In this section, the analysis of the material in the initial linear portion of the stress-strain diagram is discussed. The methods and equations, given in References 1 and 2, are summarized here for continuity. The basic parts of the analysis are

1. the thermoelastic behavior of the unidirectional lamina,
2. the transformation of the lamina properties to a given wrap angle, and
3. the superposition of internal stresses to create strain compatibility in a balanced layup.

A unidirectional lamina is shown in Figure 2 with the L, N, and T material axes. This lamina is assumed to be homogeneous and orthotropic in the L, T plane. Only stresses and strains in the L, T plane are considered; this is compatible with "thin shell theory."

Figure 2. Unidirectional lamina

The stress-strain relationships for the combined stress states are given in Eqs. (1).

$$\epsilon_L = \frac{1}{E_L} (\sigma_L - \nu_{LT}\sigma_T) + \alpha_L\Theta$$

$$\epsilon_T = \frac{1}{E_T} (\sigma_T - \nu_{TL}\sigma_L) + \alpha_T\Theta$$

$$\gamma_{LT} = \tau_{LT}/G_{LT} \quad (1)$$

Due to orthotropy of the lamina, the reciprocal relationship given in Eq. (2) results.

$$\nu_{LT}E_T = \nu_{TL}E_L \quad (2)$$

Inversion of Eqs. (1) results in expressions for the stresses.

$$\sigma_L = \frac{E_L}{(1 - \nu_{LT}\nu_{TL})} [(\epsilon_L - \alpha_L\Theta) + \nu_{TL}(\epsilon_T - \alpha_T\Theta)]$$

$$\sigma_T = \frac{E_T}{(1 - \nu_{LT}\nu_{TL})} [(\epsilon_T - \alpha_T\Theta) + \nu_{LT}(\epsilon_L - \alpha_L\Theta)]$$

$$\tau_{LT} = G_{LT}\gamma_{LT} \quad (3)$$

The thermoelastic behavior of a free, rotated lamina is obtained by transformation of Eqs. (1) and (3). Figure 3 shows the rotated unidirectional lamina with both the material and structure axes.

Figure 3. Rotated unidirectional lamina

The stress-strain relationships for the free, rotated unidirectional lamina are given by the following:

$$\begin{aligned}\epsilon_1 &= \frac{\sigma_1}{E_1^f} - \nu_{21}^f \frac{\sigma_2}{E_2^f} - \frac{T_{12}}{E_L} m_1 + \alpha_1^f \Theta \\ \epsilon_2 &= \frac{\sigma_2}{E_2^f} - \nu_{12}^f \frac{\sigma_1}{E_1^f} - \frac{T_{12}}{E_L} m_2 + \alpha_2^f \Theta \\ \gamma_{12} &= \frac{T_{12}}{G_{12}^f} - \frac{\sigma_1 m_1}{E_L} - \frac{\sigma_2 m_2}{E_L} - \alpha_{12}^f \Theta\end{aligned}\quad (4)$$

where

$$E_1^f = \frac{E_L}{\left[\cos^4 \psi + E_L \sin^4 \psi / E_T + \sin^2 2\psi (E_L / G_{LT} - 2 L_T) / 4 \right]}$$

$$E_2^f = \frac{E_L}{\left[\sin^4 \psi + E_L \cos^4 \psi / E_T + \sin^2 2\psi (E_L / G_{LT} - 2\nu_{LT}) / 4 \right]}$$

$$G_{12}^f = \frac{E_L}{\left[1 + 2\nu_{LT} + E_L / E_T - \cos^2 2\psi (1 + 2\nu_{LT} + E_L / E_T - E_L / G_{LT}) \right]}$$

$$\nu_{12}^f = E_1^f \left[\nu_{LT} - \frac{\sin^2 2\psi}{4} \left(1 + 2\nu_{LT} + \frac{E_L}{E_T} - \frac{E_L}{G_{LT}} \right) \right] / E_L$$

$$\nu_{21}^f = \nu_{12}^f \frac{E_2^f}{E_1^f}$$

$$m_1 = \sin 2\psi \left[\nu_{LT} + \frac{E_L}{E_T} - \frac{1}{2} \frac{E_L}{G_{LT}} - \cos^2 \psi \left(1 + 2\nu_{LT} + \frac{E_L}{E_T} - \frac{E_L}{G_{LT}} \right) \right]$$

$$m_2 = \sin 2\psi \left[\nu_{LT} + \frac{E_L}{E_T} - \frac{1}{2} \frac{E_L}{G_{LT}} - \sin^2 \psi \left(1 + 2\nu_{LT} + \frac{E_L}{E_T} - \frac{E_L}{G_{LT}} \right) \right]$$

$$\alpha_1^f = \alpha_L \cos^2 \psi + \alpha_T \sin^2 \psi$$

$$\alpha_2^f = \alpha_L \sin^2 \psi + \alpha_T \cos^2 \psi$$

$$\alpha_{12}^f = 2(\alpha_T - \alpha_L) \sin \psi \cos \psi \quad (5)$$

The behavior of a balanced layup can be obtained by using Eqs. (4). Consider two layers, one oriented at a positive wrap angle and the other oriented at a negative wrap angle (Figure 4). By assuming that the two lamina are rigidly bonded together, strain compatibility between layers must occur and will result in an internal shear given by Eq. (6).

$$\tau_{12}^c = G_{12}^f \left(\frac{m_1 \sigma_1}{E_L} + \frac{m_2 \sigma_2}{E_L} + \alpha_{12}^f \theta \right) \quad (6)$$

Figure 4. Balanced laminate

The strain relationships for the balanced layup as given in Reference 1 are:

$$\epsilon_1 = \frac{1}{E_1} (\sigma_1 - \nu_{12}\sigma_2) + \alpha_1\theta$$

$$\epsilon_2 = \frac{1}{E_2} (\sigma_2 - \nu_{21}\sigma_1) + \alpha_2\theta$$

$$\gamma_{12} = \tau_{12}/G_{12} \quad (7)$$

where:

$$E_1 = \frac{E_1^f}{1 - m_1^2 G_{12}^f E_1^f / E_L^2}$$

$$E_2 = \frac{E_2^f}{1 - m_2^2 G_{12}^f E_2^f / E_L^2}$$

$$\nu_{12} = E_1 \left[\nu_{12}^f / E_1^f + m_1 m_2 G_{12}^f / E_L^2 \right]$$

$$\nu_{21} = \nu_{12} E_2 / E_1$$

$$G_{12} = \frac{(1 - \nu_{12}^f \nu_{21}^f) G_{12}^f}{1 - \nu_{12}^f \nu_{21}^f - \frac{m_1 (m_1 + \nu_{21}^f m_2) E_1^f G_{12}^f}{E_L^2} - \frac{m_2 (m_2 + \nu_{12}^f m_1) E_2^f G_{12}^f}{E_L^2}}$$

$$\alpha_1 = \alpha_1^f - \alpha_{12}^f m_1 G_{12}^f / E_L$$

$$\alpha_2 = \alpha_2^f - \alpha_{12}^f m_2 G_{12}^f / E_L \quad (8)$$

Stresses can be obtained by inverting Eqs. (7) as shown in Eqs. (9).

$$\sigma_1 = \frac{E_1}{(1 - \nu_{12}\nu_{21})} \left[(\epsilon_1 - \alpha_1\theta) + \nu_{21}(\epsilon_2 - \alpha_2\theta) \right]$$

$$\sigma_2 = \frac{E_2}{(1 - \nu_{12}\nu_{21})} \left[(\epsilon_2 - \alpha_2\theta) + \nu_{12}(\epsilon_1 - \alpha_1\theta) \right]$$

$$\tau_{12} = G_{12}\gamma_{12} \quad (9)$$

The balanced laminate behaves as an orthotropic material with the planes of orthotropy oriented along the structure axes. By assuming that the material is a series of balanced laminates and is symmetrical throughout the thickness, the gross material behavior is given by Eqs. (7) and (9).

Prediction of Yield Point - Region Two

The "yield point" (Figure 1) is determined by failure of an individual lamina. Tensile stress normal to the fiber direction and shear stress in the matrix are the primary causes of lamina failure. The modified Hill Failure Criterion used to define yield level (YL), given in References 1 and 3, is redefined in Eq. (10) for continuity.

$$YL = \left(\frac{\sigma_L}{F_L} \right)^2 - \left(\frac{\sigma_L \sigma_T}{F_L^2} \right) + \left(\frac{\sigma_T}{F_T} \right)^2 + \left(\frac{\tau_{LT}}{F_{LT}} \right)^2 \quad (10)$$

The yield point occurs when the yield level reaches 1.0. F_L and F_T are either tensile or compressive strengths, depending upon the sign of the lamina stresses σ_L and σ_T .

The lamina stresses in the principal orthotropy directions are obtained by transformation of the stresses in the structure axes as shown in Eqs. (11).

$$\begin{aligned}\sigma_L &= \sigma_1^f \cos^2 \psi + \tau_{12}^f \sin 2\psi + \sigma_2^f \sin^2 \psi \\ \sigma_T &= \sigma_2^f \cos^2 \psi - \tau_{12}^f \sin 2\psi + \sigma_1^f \sin^2 \psi \\ \tau_{LT} &= \tau_{12}^f \cos 2\psi + \frac{1}{2} (\sigma_2^f - \sigma_1^f) \sin 2\psi\end{aligned}\quad (11)$$

where:

$$\begin{aligned}\sigma_1^f &= \sigma_1 \\ \sigma_2^f &= \sigma_2 \\ \tau_{12}^f &= \tau_{12} + G_{12}^f \left(\frac{m_1 \sigma_1}{E_L} + \frac{m_2 \sigma_2}{E_L} + \alpha_{12}^f \theta \right)\end{aligned}\quad (12)$$

Post Yield Behavior

After matrix yield occurs, as defined in the previous section, the material can still carry a load in the direction of the fibers. This portion of the stress-strain curve is represented by using a modification of the "Netting Analysis" methods discussed in Reference 3.

Consider two layers oriented at positive and negative wrap angles. Assume that no load can be transmitted in the plane of the laminate except parallel to the fibers. A free body of one of these layers is shown in Figure 5.

Figure 5. Free body diagram

Equilibrium in the 1 direction dictates that:

$$\sigma_f = \frac{\sigma_1}{\cos^2 \psi} \quad (13)$$

Equilibrium in the 2 direction must be supplied by a shear stress transmitted between layers:

$$\sigma_E = \sigma_f \sin^2 \psi = \sigma_1 \frac{\sin^2 \psi}{\cos^2 \psi} \quad (14)$$

This equilibrium stress must exist for this model to be valid. This force balance may occur either through frictional interference at the filament cross-over points or through some interlaminar shear compatibility in the matrix.

If no lateral motion exists at the filament cross-over points, the total elongation in the fiber is:

$$\delta = L\epsilon = L \frac{\sigma_f}{E_f} \quad (15)$$

Converting deflections and stresses into the structure axes system ($\underline{1}$ direction), the equivalent slope of the stress-strain curve is:

$$E = E_L \cos^4 \psi \quad (16)$$

Equation (16) was derived by assuming that no lateral motion exists at the filament cross-over points. In actual material, some motion will occur, causing a reduction of this slope. At the extreme matrix deformation, the slope will approach zero.

Prediction of Ultimate Strength

In the previous section, the stress and deformation patterns based on "Netting Analysis" techniques were discussed. This behavior can exist if two conditions are met:

1. if the stress in the fiber direction does not exceed the ultimate fiber strength and
2. if a transverse equilibrium stress is present because of filament cross-over friction on interlaminae matrix shear.

Ultimate tensile strength will be defined as the stress which exceeds either of these two conditions. Mathematically, the first condition can be expressed by Eq. (17).

$$\frac{\sigma_1}{\cos^2 \psi} \geq UTS_f \quad (17)$$

For the second condition no mathematical expression now exists that relates this failure mode to the fiber or matrix properties. This failure mode will result in very large deformations before complete failure (sometimes referred to as "bulging" in cylinder tests).

Residual Stresses

In contrast with homogeneous material, the material considered here is composed of various balanced layers which create internal stresses due to a uniform temperature change. Considering a material with no external stress and only a uniform temperature change, Eqs. (11) and (12) can be rewritten to define the lamina stress patterns:

$$\sigma_1^f = 0$$

$$\sigma_2^f = 0$$

$$\tau_{12}^f = G_{12}^f \alpha_{12}^f \theta \quad (18)$$

and

$$\sigma_L = G_{12}^f \alpha_{12}^f \theta \sin 2\psi$$

$$\sigma_T = -\sigma_L$$

$$\tau_{LT} = G_{12}^f \alpha_{12}^f \theta \cos 2\psi \quad (19)$$

Since large temperature extremes occur during the manufacture of carbon or graphite filament wound materials, it is very important to account for lamina stresses due to temperature changes. By assuming a manufacturing temperature at which the stresses are essentially zero, the residual stresses occurring after reduction to room temperature can be calculated by Eqs. (18) and (19).

Thermal Stress Evaluation

Since we assume that the layup is always balanced and symmetric through the thickness, the material behavior is characterized by Eqs. (7) and (9), which show that the gross material behavior is the same as that of a homogeneous orthotropic material with the planes of orthotropy oriented along the structure axes.

Consider the long, thin cylindrical shell shown in Figure 6.

Figure 6. Filament wound cylindrical shell

The forcing function of interest is a temperature gradient through the shell thickness, uniform at all circumferential and longitudinal locations (Figure 7).

Figure 7. Temperature distribution

Because of thin shell approximations, the solution to this problem can be obtained by forcing constant strain in the 1 direction and the 2 direction and maintaining a zero force resultant in both directions. Mathematically this is expressed:

$$\epsilon_1 = c_1$$

$$\epsilon_2 = c_2$$

$$\int_0^h \sigma_1 d\eta = 0$$

$$\int_0^h \sigma_2 d\eta = 0 \quad (20)$$

Example 1

Material Properties for CY2-5 Fiber Composite

In this section the methods presented in the previous sections are used to predict material behavior for a material made from CY2-5 fibers, a CVD matrix and heat-treated at 3000°C. Test data at room temperature are available for this material at various wrap angles. Some thermal expansion data is available up to 3000°F.

The lamina properties used for this example are:

$$E_L = 4 \times 10^6 \text{ psi}$$

$$E_T = 1.8 \times 10^6 \text{ psi}$$

$$G_{LT} = 1.0 \times 10^6 \text{ psi}$$

$$\mu_{LT} = 0.52$$

$$\alpha_L = 1.7 \times 10^{-6}/^\circ\text{F}$$

$$\alpha_T = 3.5 \times 10^{-6}/^\circ\text{F}$$

$$F_L \text{ (tension)} = 13 \times 10^3 \text{ psi}$$

$$F_L \text{ (compression)} = 13 \times 10^3 \text{ psi}$$

$$F_T \text{ (tension)} = 6 \times 10^3 \text{ psi}$$

$$F_T \text{ (compression)} = 12 \times 10^3 \text{ psi}$$

$$F_{LT} = 6 \times 10^3 \text{ psi}$$

Using Eqs. (5) and (8) the effective thermoelastic constants for a balanced wrap configuration for several wrap angles are given in Table 1.

TABLE 1
Thermoelastic Constants

Wrap Angle (Degrees)	0	20	30	45
E_1 (psi $\times 10^6$)	4.0	3.67	3.32	2.74
E_2 (psi $\times 10^6$)	1.8	1.99	2.24	2.74
ν_{12}	0.52	0.49	0.45	0.37
ν_{21}	0.23	0.26	0.30	0.37
G_{12} (psi $\times 10^6$)	1.0	0.97	0.95	0.94
α_1 ($\times 10^{-6}/^{\circ}\text{F}$)	1.7	1.82	1.97	2.34
α_2 ($\times 10^{-6}/^{\circ}\text{F}$)	3.5	3.16	2.83	2.34

The residual stress pattern which occurs after return from graphitization temperature to room temperature is based on a uniform temperature drop of 2700°F . This temperature drop was used in order to match room temperature test data as well as possible. Equations (19) give the lamina residual stresses for $\theta = 2700^{\circ}\text{F}$. Table 2 gives the residual stresses for several wrap angles.

TABLE 2
Lamina Residual Stresses

Wrap Angle (deg)	0	20	30	45
σ_L (psi)	0	-2000	-3500	-4550
σ_T (psi)	0	2000	3500	4550
T_{LT}	0	-2300	-2000	0

The room temperature material behavior is calculated by using the above data and Eqs. (10), (16), and (17). Predicted uniaxial stress-strain curves are compared to test data for various wrap angles in Figures 8 and 9.

Figure 8. Stress-strain curves for 20° wrap angle material

Figure 9. Stress-strain curves for 45° wrap angle material

The elevated temperature stress-strain curves can be calculated in the same manner, except that the temperature used to obtain lamina stresses from Eqs. (19) must be obtained by subtracting the residual stress temperature (2700°F) from the temperature of concern (i.e., to obtain the stress strain relationship at 3000°F, $\theta = 3000 - 2700$). A typical elevated temperature stress-strain curve is shown in Figure 10.

Figure 10. Elevated temperature stress-strain curve

Note that the curve is completely linear. This results from the fact that the ultimate tensile stress obtained from Eqs. (17) is lower than the yield criterion obtained from Eqs. (10). Unfortunately, test data at elevated temperatures are not available to provide correlation with this theory. This elevated temperature stress-strain behavior is based on the assumption that the lamina properties given earlier are independent of temperature. The elevated-temperature behavior can also be calculated by assuming that the elastic and yield properties vary as a function of temperature as shown in Figure 11. A typical stress-strain curve is shown in Figure 12 assuming a temperature dependence of the lamina properties.

Figure 11. Temperature effects on lamina properties

Figure 12. Elevated temperature stress-strain curve

Example 2

Thermal Stresses for Cy2-5 Fiber Composite

Equations (20) are used to determine the stress levels due to a given thermal environment through a cylindrical shell. Using the temperature profile given in Figure 13, the stresses in the structure axes (σ_1 and σ_2) were calculated for a 20-degree wrap angle composite cylinder. The tensile stresses on the inner surface are given for both constant and temperature-dependent lamina properties in Table 3.

Figure 13. Typical temperature profile

TABLE 3
Calculated Thermal Stresses

$\sigma_1 = 10670 \text{ psi}$	}	Constant Lamina Properties
$\sigma_2 = 8850 \text{ psi}$		
$\sigma_1 = 9080 \text{ psi}$	}	Temperature Dependent Lamina Properties
$\sigma_2 = 7530 \text{ psi}$		

The effects of temperature dependence of the lamina are very critical in predicting the thermal stress levels.

The stresses in the structure axis directions and the temperature are used in Eqs. (10) and (11) to determine lamina stresses and yield levels. The results of these calculations are given in Table 4.

TABLE 4
Lamina Stresses and Yield Levels

	Constant Lamina Properties	Temperature Dependent Lamina Properties
σ_L (psi)	11,200	9,470
σ_T (psi)	8,320	7,140
T_{LT} (psi)	300	190
Failure Level	2.12	1.12

Concluding Comments

The structural analysis methods presented here for use with carbon-carbon filament wound material give satisfactory results for calculations of mechanically induced loads at room temperature. It is felt that these methods will accurately predict elevated-temperature properties and thermal stresses. Full-scale reentry-vehicle heatshields have been analyzed, but lack of high-temperature material data has been a limitation.

In the future, these methods will be expanded to solve more complex structural problems. The lack of experimental verification and material properties at high temperatures can be corrected by future testing of this filament wound material.

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**A THERMOSTRUCTURAL SENSITIVITY ANALYSIS
FOR GRAPHITIC NOSE TIPS***

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ABSTRACT

In order that some priority can be given as to which property is the most "design influencing", a material property sensitivity study was performed on a graphitic nose tip. The philosophy involved in this was to vary the magnitude and/or the trend of a particular property with temperature consistent with the variation evident in measured design data.

The sensitivity study was conducted in two parts. In the first, the temperature gradients were assumed invariant and the effect of variable mechanical properties were studied. In the second part, the mechanical properties were assumed invariant and effect of variable thermal parameters was studied. The input variables to the sensitivity analysis are those which are used to predict the ablation characteristics, temperature profiles and the state of stress of the re-entering body. The specific material properties which were varied are elastic moduli (with and across grain), shear modulus, Poisson's ratio, thermal expansion coefficient (with and across grain), thermal conductivity, total hemispherical emittance, and specific heat (high temperature behavior).

In addition to an absolute dependence on strain to failure, the results demonstrate primary reliance on thermal coefficient of expansion, thermal conductivity and elastic modulus trend with temperature.

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INTRODUCTION

A detailed thermostructural analysis of a graphitic re-entry vehicle nose tip requires a large number of material properties, the actual number depends on the anisotropy of the material. For a given flight environment, the aerodynamic heating is used to predict the ablation performance and resulting in-depth temperature gradients, which in turn are used to predict the state of stress within the nose tip. These predictions depend on the material property data input to the computer programs.

In order that some priority can be given to the test matrix as to which property is the most design influencing, a material property sensitivity study was performed on a graphitic nose tip. The philosophy involved was to vary the magnitude and/or the trend of a particular property with temperature. The comparison criterion used was the effect on the predicted thermal stress (strain) and the corresponding margin of safety, i.e., actual stress (strain) capability compared to predicted stress (strain).

The model chosen for this study was a nose tip designed and flown by GE-RESO on a recent RVTO flight. The approach used in performing this study considered the following:

- A. A representative nose tip configuration is studied.
- B. The material anisotropy is treated, in this case transversely isotropic, e.g., a molded graphite such as ATJ-S.
- C. Material property variations with temperature are selected from actual design data. In this case, an attempt was made to select curves consistent with the degree to which specific properties are known and measured.
- D. The sensitivity is measured by the perturbation to the predicted strain and margin of safety which is the final measure of design integrity for an actual re-entry analysis.

The nose tip studied is a typical blunt nose configuration and is shown in Figure 1. The material was made from the Union Carbide molded-to-shape ATJ-S graphite.

MATERIAL PROPERTY VARIATION

The sensitivity study was conducted in two parts. The first assumed invariant temperature gradients obtained from the thermal program and computed the effect of variable material and structural properties upon the margin of safety. The second assumed invariant structural properties and computed the effect of variable thermal parameters upon the margin of safety. The predictions were performed for the critical thermostructural altitude of this particular nose tip.

The input variables to the sensitivity analysis are those which are used to:

- A. Predict the ablation characteristics and temperature profiles.
- B. Predict the state of stress of the re-entering body.

The specific material properties which were varied are:

- A. Structural Properties
 - 1. Elastic moduli (with and across grain)
 - 2. Shear modulus

3. Poisson's ratio
4. Thermal expansion coefficient (with and across grain)

B. Thermal Properties

1. Thermal conductivity
2. Total Hemispherical Emittance
3. Specific heat (high temperature behavior)

Elastic Moduli

The input data for elastic moduli attempt to cover the regular spread of data which is found experimentally on graphitic materials. When this is the case, the maximum recorded moduli curve is chosen for design, although this is not the most expected value. Analyses which have used this data, however, are therefore conservative or tending to the worst case condition. The data used in this analysis was generated for a molded to shape ATJ-S material. The experimental techniques and facilities used in obtaining this data are discussed in Reference 1. The experimental data points and the sensitivity curves are shown in Figure 2 and Figure 3 for with grain and across grain data, respectively. Three input curves are shown for each direction representing a maximum value trend with temperature (design or nominal values), a minimum (highly optimistic) and an intermediate value trend. In the analyses the two equivalent trend lines for with and across grain data were used together, i.e., the maximum modulus versus temperature for with grain and across grain directions.

Thermal Expansion Coefficient

The thermal expansion data used in the analysis is again based upon experimental data. These are shown in Figure 4 and Figure 5 for with grain and across grain, respectively. The data again attempts to cover ranges of experimental accuracy associated with elevated temperature thermal expansion measurements. The lower curves represent the design data obtained on the particular graphite material¹, while the other curves have been produced by multiplying the value obtained at 5000°F by the ratios 1.05, 1.10 and 1.20, respectively and fairing in the curves from 2500°F. In this way they represent 5, 10, and 20 percent variations in the elevated temperature expansion behavior of the material. Differences of this order are by no means uncommon in experimental data for graphitic and ceramic materials and represent a combination of material variability and experimental inaccuracy (probably independent of test method at $\approx 5000^\circ\text{F}$ temperature) which is difficult to separate. As in the example of elastic moduli the two equivalent trend lines for with and across grain data were used together in the analyses.

Shear Modulus

The shear modulus for an isotropic material can be calculated using the elastic modulus and Poisson's ratio, since, for an isotropic material there are only two independent elastic constants. This is also true for the symmetry plane in the transversely isotropic case of a molded graphite. However, the shear modulus between the with grain and across grain directions still requires measurement. It was assumed for this analysis that the shear modulus had the same value as the isotropic plane calculated value. These values as a function of temperature are shown in Figure 6 together with shear modulus data reduced by 20 and 50 percent of the original curves. The data has been calculated using the elastic moduli from Figure 2 and Figure 3, and the constant Poisson's ratio of 0.28 shown in Figure 7.

Poisson's Ratio

The effect of variations in Poisson's ratio has been evaluated using the data sets shown in Figure 7. This data attempts to evaluate two features; the absolute value of Poisson's ratio, and the trend with temperature.

The data is essentially in two sets, a constant trend with temperature and a variable trend with temperature. Within each set, there are two absolute levels of data such that in comparing the absolute levels between all data, there can be a 100 percent difference in Poisson's ratio value between the upper constant level and the lower variable level at the critical temperature of 3000°F.

In this study, the Poisson's ratios have been given the following convention:

ν_{ww} = Ratio of lateral with grain strain to axial strain of a specimen loaded with the grain.

ν_{wa} = Ratio of lateral with grain strain to axial strain of a specimen loaded across the grain.

ν_{aw} = Ratio of lateral across grain strain to axial strain of a specimen loaded with the grain

$$\text{and} \quad \frac{\nu_{aw}}{\nu_{wa}} = -\frac{E_w}{E_a}$$

Thermal Conductivity

Direct measurements have been made at GE-RESO on representative ATJ-S billets under the RVTO program so that firsthand experimental data were available for this already quite well characterized graphite.

The recommended variations about the nominal thermal conductivity functions (the experimentally measured values) were ± 20 and ± 50 percent. The nominal design data are given as Table 1. Variations on the order of ± 20 percent ($0.8k_0$ and $1.2k_0$) are the most probable and would conservatively correspond to values bound by the standard deviation (one sigma) of the experimental precision and accuracy. This is based on observation of the measurements at GE, standardization of GE thermal conductivity instruments with other graphite laboratories and also other laboratories' measurements on ATJ graphite.

The 50 percent variation ($1.5k_0$ and $0.5k_0$) would correspond to a high confidence range of certainty for ATJ thermal conductivity. This margin was a quite realistic assumption for graphites up to two or three years ago when multiple sources of high temperature measurements and a general improvement in the techniques and sample characterization occurred. This development can be observed in the Proceedings of the Thermal Conductivity Conferences and in the TPRC volumes, where some of the earlier measurements on ATJ are not even characterized as to grain orientation or density. Such deficiencies are not likely for a material for which a thermal and electrical conductivity of 1.3 to 1 have been measured and for which a three percent change in conductivity occurs for a one percent change in density over a range of densities from 1.69 to 1.83 gm/cc.

Emittance (Total Hemispherical)

The possible variations to be anticipated in this parameter are prospectively among the widest of all the thermophysical properties of polycrystalline graphite. This is despite the well known reproducibility of the machined surface emittances of unalloyed polycrystalline graphites at room temperature. However, it has been shown at GE-RESO that even at room temperature, exceptionally fine machined finishes can be made to produce a wide range of room temperature emittances. In particular for ATJ grades, it was shown that a sample polished to 20 microinch RMS finish had a total normal emittance of 0.5 as compared to 0.8 for an 80 RMS finish on identical material, and the nominal figure of 0.9 for shop finished and handled surfaces. The effects of aerodynamic friction and

Table 1. Thermal Property Sensitivity Analysis – Thermal Properties
Nominal Values – ATJ-S Graphite

Temperature (°R)	C_p^* (Btu/lb °R)	K (Btu/sec ft °R)	ϵ_H	C_p^{**} (Btu/lb °R)
1	.01	.03	.9	.01
460	.11	.026	.9	.11
560	.17	.0244	.9	.17
660	.215	.023	.9	.215
760	.247	.0217	.9	.247
860	.275	.0205	.9	.275
960	.3	.0194	.9	.3
1460	.387	.0148	.9	.387
1960	.44	.0114	.9	.40
2460	.472	.0092	.9	.416
2960	.49	.0078	.9	.475
3460	.498	.0068	.9	.492
5000	.519	.006	.9	.525
9000	.6	.006	.9	1.0
20000	.7	.006	.9	1.0
*JANAF Data ²				
**Rasor and McClelland Data				

ablation chemistry are an obvious complication to this uncertainty in the graphite R/V surface emittance - and an uncertainty that will not be clarified by laboratory measurements in the foreseeable future because of the necessity of reproducing the specific re-entry aerodynamic and chemical environment.

Accordingly, perturbed values of 0.8, 0.7 and 0.5 were used for the thermodynamic sensitivity analyses; to be compared to the nominal design value of 0.90 ± 0.05 .

Specific Heat

There has been a long standing controversy over the nature of the specific heat function of graphite at the highest temperatures, approaching the solid-vapor phase diagram interface. The results of the well known 1956 study by Rasor and McClelland³ are shown in Figure 8. The sudden, exponential increase in this specific heat function in the temperature range 3000 to 3500°C is a drastic departure from the JANAF data² used for design purposes. The two are compared in the Figure 8.

The Rasor and McClelland data, and the experimental technique used to determine it, constitute a continuing controversy in high temperature graphite technology. Unfortunately, no rival techniques have since been developed to corroborate or refute their data.

Accordingly, this variation from the nominal design data was chosen for a third thermophysical parameter, related to the graphite ablation surface phenomenology.

RESULTS

Structural Property Variations and Their Effect On Thermal Stress Predictions

The critical stress state for graphite nose tips is in the transition region between the stagnation region of the nose tip and the nose tip skirt, Figure 1. Structural analysis indicates that the critical stress state in a nose tip occurs under the following conditions:

- A. Material whose temperature is below 3000°F.
- B. Material in a biaxial state of stress in the tension-tension quadrant.
- C. Material on or adjacent to a free surface.

Elastic Modulus, Poisson's Ratio, Shear Modulus and Thermal Expansion Coefficient

Thermal strains and the corresponding margins of safety have been predicted for variations in E , ν , $\Delta L/L$, and G .

$$\text{M.S.} = \frac{\epsilon_{\text{ult}}}{\epsilon_{\text{calc}}} - 1$$

In this case the allowable strain used to calculate the margins of safety was that obtained experimentally for the molded to shape ATJ-S material used in the RVTO re-entry vehicle. This data is presented in Figure 9 and Figure 10. The temperature profiles in the nose at the critical re-entry altitude are shown in Figure 11. These were obtained for the nominal thermal property data.

The predicted margin of safety obtained under these conditions for variations in elastic modulus, Poisson's ratio, shear modulus and thermal expansion coefficient are shown in Figure 12 for both the hoop direction and the R-Z plane.

Thermal Conductivity, Specific Heat and Emittance

The sensitivity analysis to evaluate the effect of thermal parameters was conducted in the skirt region of the nose tip at the station shown in Figure 1. In this analysis the radial temperature distribution resulting from the thermal data variations are used in an anisotropic generalized plane strain cylinder analysis. The analysis was performed at three different altitudes (60, 55 and 49 K ft.); a range which includes the critical thermostructural altitude. The temperature profiles using the nominal thermal property data are shown in Figure 13.

The thermal stress margins of safety for these variables are shown in Figure 14. It can be seen that this area of the nose is not a critical one by the relatively large values of margin of safety as compared to the critical region considered for the structural variables.

CONCLUSIONS

It is apparent from an examination of the results of the sensitivity analysis for thermal stress, that the dominant input parameters for the theoretical predictions are thermal expansion coefficient, elastic modulus, and thermal conductivity. Poisson's ratio, shear modulus and emittance have a less marked effect. It also does not appear to be important which data set is used concerning specific heat.

It would be extremely difficult to separate elastic modulus and thermal expansion coefficient as to which has the greater effect on the predicted level of thermal stress since both are sensitive parameters. Experimentally both measurements require extremely accurate dimensional measurements

at elevated temperatures, e.g., an error of +10 percent for ATJ-S graphite at 5000°F is only a change of the order of 0.0015 in/in strain measurement while a 10 percent error in determining strain for a modulus determination at 2000 psi (approximately 100 μ inch) is sufficient to change the modulus from 2.0×10^6 psi to 1.8×10^6 psi. Actually the range of data covered for the with grain ATJ-S graphite used in the present study reflects moduli variations at 3000°F of 1.5 to 2.0×10^6 psi corresponding to only 333 μ inch of strain. Errors of this order are close to the state-of-the-art with respect to optical measurement techniques in conjunction with graphite material behavior.

A higher conductivity indicates a larger margin of safety – the positive 20 percent tolerance raises the margin from the nominal 164 percent to 199 percent. But the equally probable minus 20 percent tolerance drops the margin to 109 percent. There are two primary indications from these results: 1) that scrupulous care must be exercised in the use of lower-bounded graphite thermal conductivity data, and 2) that effort toward the achievement of reliable, routine experimental techniques for assuring a ± 10 percent margin on this thermophysical parameter is immediately justified.

Shear modulus and Poisson's ratio are relatively insensitive variables. A 20 percent change in the shear modulus produces a three percent change in axial strain. For the most part, variations in Poisson's ratio will not significantly alter the results of a nose tip thermostructural analysis. This is shown for both the constant with temperature trends and the variable with temperature trends. In comparing absolute value for example, at a temperature of 3000°F, the difference between Poisson's ratio is of the order of 100 percent for the input data considered, e.g., the highest constant value with temperature and the lowest variable with temperature. The difference in margin of safety represents only three percent at maximum, i.e., 9.1 percent to 7.2 percent for hoop margins and 10.2 percent to 7.1 percent in the R-Z margins. Consequently one may presume an extremely low dependence for Poisson's ratio.

A change in the value of emissivity from 0.9 to 0.5 produces a change in the margin of safety from 164 percent to 138 percent or approximately - 16 percent. If a similar effect was shown in the critical region of the nose tip this would correspond to a change in the margin of safety of say 10 percent to 8.6 percent, a change which could easily be accounted for by a relatively small change in thermal expansion coefficient or elastic modulus trend.

The choice as to which specific heat trend with temperature is equally insignificant, since it results in margin of safety changes less than those due to the emissivity changes.

All of the above variables considered are inputs to the prediction programs for thermal stress and as such, their effect is shown in obtaining the predicted strain output which may be compared to the actual strain capability of the material. In computing the margins of safety, it is the actual strain to failure obtained experimentally which is used as the strain comparator. Any change, therefore, in the strain to failure for the material would have a highly significant effect on thermal stress margin of safety. It becomes important, therefore, that the minimum strain to failure is known accurately.

The data presented above has been referenced to a margin of safety criterion from which it has been shown that the most sensitive thermal stress influencing parameters are elastic modulus, thermal expansion coefficient, and thermal conductivity with strain to failure as the comparator. An alternate method of presenting this data is with reference to thermal strain as shown in Figures 15 through 17, thermal expansion coefficient, elastic modulus and thermal conductivity. It must however be appreciated that the trends of properties used in these analyses are dependent upon estimates of the scatter of data which could occur in a given graphite billet (due to test method and material variability). If elastic modulus data has been proportionately varied in the analysis i.e., a 30 percent reduction or increase across the temperature range of interest, no effect would have been seen on thermal strain.

As a result of the thermal stress sensitivity analysis some order of priority may be given to the data required in the analyses. This is shown below:

Data Used in Thermal Stress Analysis for Graphite Nose Tips*

Structural Data		Thermal Data	
Elastic Modulus**	1	Thermal conductivity	1
Thermal expansion coefficient	1	Emissivity	2
Poisson's ratio	2	Specific heat	2
Shear Modulus	2		
Strain to Failure	1		
*Number indicates the order of priority for data accuracy.			
**Elastic Modulus trend with temperature.			

The fact that a particular data point is given a secondary priority does not presume that the data is not needed, but only indicates a secondary ranking on accuracy. Since a similar analysis is used for both polycrystalline graphite and carbon-carbon systems this sensitivity analysis would apply to both types. Throughout all these analyses tensile data has been used in lieu of compressive data.

A summary of the sensitivity studies which were conducted is illustrated by Figure 18.

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Fig. 1. Nose Tip Model.

Fig. 2. Material Sensitivity - Modulus of Elasticity vs Temperature, With Grain.

Fig. 3. Material Sensitivity - Modulus of Elasticity vs Temperature, Across Grain.

Fig. 4. Material Sensitivity - Thermal Expansion vs Temperature, With Grain.

Fig. 5. Material Sensitivity - Thermal Expansion vs Temperature, Across Grain.

SHEAR MODULUS, PSI X 10⁵

Fig. 6. Material Sensitivity - Shear Modulus vs Temperature.

Fig. 7. Material Sensitivity - Poisson's Ratio vs Temperature.

Fig. 8. Specific Heat of All Graphites.

Fig. 9. Special Processed ATJ-S Graphite, Maximum Allowable Tensile Strain, With Grain.

Fig. 10. Special Processed ATJ-S Graphite, Maximum Allowable Tensile Strain, Across Grain.

Fig. 11. Nose Tip Isotherms at Critical Altitude.

Fig. 12. Predicted Thermal Stress Margin of Safety for Structural Property Variations.

Fig. 13. Temperature Profiles, Station 3.0.

Fig. 14. Predicted Thermal Stress Margin of Safety for Thermal Property Variations, Skirt Region of Nose Tip.

Fig. 15. Sensitivity of Thermal Strain to Thermal Expansion $(\Delta L/L)$.

Fig. 16. Sensitivity of Thermal Strain to Modulus.

Fig. 17. Sensitivity of Thermal Strain to Conductivity.

Fig. 18. Material Property Sensitivity Analysis Summary.

FRACTURE

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THE DEFORMATION AND FRACTURE MECHANISMS
IN POLYCRYSTALLINE GRAPHITES

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ABSTRACT

Manufactured polycrystalline graphites are brittle solids which normally contain from 15 to 30% of void volume in the form of cracks, shrinkage cavities, and blowholes. In mechanical analyses, however, they are assumed to be structurally continuous and capable both of plastic deformation and of strain-hardening. Justification for these assumptions is found in the unique combination of crystal structure and defect structure that exists in a polycrystalline graphite. This structural situation permits microcracks to form at small "background" defects, extend over short distances, and stop, after which --at higher stress -- additional cracks form elsewhere and undergo similar cycles. The dilatation represented by crack opening produces macroscopic strain and, if the volume increase is taken into account, it can be treated in the same terms used for plastic deformation. The requirement for higher stress to produce each successive generation of cracks is the equivalent of strain-hardening. And the point-to-point load-transfer occurring when growth of one crack stops and that of another begins demonstrates the structural continuity required by continuum mechanics.

Fracture occurs when, locally, the concentration of microcracks becomes sufficient so that they overlap and join to form a discontinuous macrocrack of critical size, which then propagates at an increasing rate. Microcracks form in highest concentration in the regions of stress-concentration adjacent to large, "disparate" defects. The fracture path, therefore, extends preferentially from one large defect to the next. Between them it is controlled jointly by the stress field and by the nature and distribution of background defects, and normally it is branching and irregular.

INTRODUCTION

The microstructural changes that occur in a polycrystalline graphite as it is strained to fracture in tension have been reported for both room temperature^{1,2,3} and elevated temperature^{4,5,6} tests. In general it has been observed that, independent of temperature, microcracks approximately normal to the tension axis form at small strain, grow to a limiting size, and stop growing. Then, with increasing strain and at higher stress, new microcracks form elsewhere in the structure, grow to about this same size, and also stop, to be replaced at still higher stress by additional generations of microcracks. Eventually the microcracks become so numerous that somewhere they overlap, join, and create a fracture path. This is a brittle behavior, although -- remarkably for a brittle material -- the cracks formed early in the process in general do not propagate directly to fracture.

The unique cracking behavior of a polycrystalline graphite, and its final fracture, are controlled jointly by its crystal structure, which is well understood, and its defect structure, which is understandable in terms of the raw materials and processes used in its manufacture.

GRAPHITE MANUFACTURE

All graphites are nearly pure carbon, and it is not practical to melt and cast carbon. The polycrystalline graphites therefore are made by the common ceramic technique of mixing a solid particulate filler with a liquid binder, compacting the mixture, and heating the compact to expel volatile matter and produce the phase or phases desired in the finished body.

In the case of graphite, the filler consists of nearly pure carbon -- usually a ground, calcined, petroleum coke. Coke fillers vary widely in mean particle size, particle-size distribution, shape, shape distribution, and internal structure. With rare exceptions, however, the filler particles are rigid, irregular in shape, and incapable of packing to fill space completely. Thus, Fig. 1 is a photomicrograph of a region in a polycrystalline graphite in which (due either to poor mixing or to use of insufficient binder) only filler particles are present. Apparently bridging has occurred, and the proportion of interparticle void is abnormally great. However, from the shapes of the filler particles -- which are typical of a coke -- it is evident that these particles could not be arranged in such a way as to eliminate all of the voids between them. In fact, even with a very desirable distribution of particle sizes, it is unusual for a coke filler to pack to as much as 70% of full density. Thirty percent or more of interparticle void is normal for a packed coke filler.

In general, the graphite manufacturer tries to just fill this void volume with binder, as has been done in Fig. 2. Other binders are sometimes used, but the most common is a coal-tar pitch. To be liquid at the temperatures at which the graphite components are mixed and compacted, the binder must contain a reasonable proportion of atoms other than carbon -- principally hydrogen. During baking the binder is pyrolyzed and these heteroatoms are driven off as gases. This has three incidental effects: (1) Some of the binder carbon is lost in chemical combination with the heteroatoms, which themselves represent a significant fraction of the original binder weight. The yield of solid carbon from the binder is rarely as much as 60% of the original binder weight, and often is less than 50%. (2) As the binder loses mass it decreases in volume, producing shrinkage cracks and voids. (3) The gases evolved during binder pyrolysis either create continuous escape channels to the surface or are trapped within the compacted

body, producing blowholes if the matrix is still plastic, and large cracks if it is not.

The baking heat-treatment, then, reduces the hydrocarbon binder to solid carbon and also produces within the formed body a variety of defects, including: shrinkage cracks, shrinkage cavities, connected porosity, blowholes, and macrocracks. Some of these defects are illustrated in Fig. 3. If the filler coke was not previously calcined to some temperature equal to or higher than that used for baking, its pyrolysis will also contribute to this structural damage.

After baking, the carbon body is heat-treated to some higher temperature, usually above 2500°C, to graphitize it -- i.e., to permit the carbon atoms to arrange themselves into the layered hexagonal crystal structure of graphite. Graphitization is accompanied by additional shrinkage, due both to the atomic rearrangement and to a further loss of volatiles, and this may produce additional cracks. Finally, because of the anisotropies of thermal contraction of individual graphite crystals and their relative misorientations within and between filler particles and binder residues, there are restraints on the contraction of the crystals as they cool from the graphitizing temperature. Mrozowski stresses⁷ are developed, which may in part be relieved by formation of additional microcracks, but must also in part remain as a pattern of local residual tension and balancing local residual compression within the finished graphite body.

DEFECT STRUCTURES

As results of the chemical and physical changes that occur during its manufacture, a polycrystalline graphite contains a variety of defects which, together, ordinarily constitute about 15 to 25% of its bulk volume. A small part of this void volume represents imperfect regions between adjacent misoriented, graphite crystals. The rest of it appears as defects of two classes, which have been identified by Pears⁸ as background defects and disparate defects.

Background defects in a polycrystalline graphite are generally small relative to the sizes of filler particles, and are quite uniformly distributed throughout the graphite structures. They include small blowholes, cracks, and zones of easy cleavage within filler particles; shrinkage cracks at interfaces between filler particles and binder residue; shrinkage cracks and shrinkage cavities within the binder residue; isolated small gas pockets within the binder residue; and connected porosity through the binder residue created by gas escape. Once normal raw materials and manufacturing procedures have been selected to make a graphite, the occurrence of most of these defects is inevitable. The background defects can be increased in size and number by poor choices either of materials or of procedures, and to a degree they can be corrected by such post-manufacturing treatment as impregnation and hot-working. In a well-made, fine-grained graphite they may be too small to be resolved by an optical microscope. However, background defects are always present, and they have important effects on most of the properties of a polycrystalline graphite.

Disparate defects are much larger than background defects, and in general they could have been avoided by improving either the raw materials or the manufacturing procedures. The most common disparates, and apparently the most difficult to avoid in routine graphite production, are relatively large blowholes of the type shown in Fig. 4. These are produced by gas entrapment either during mixing of the graphite components or during the early stages of baking, while the binder is still plastic. In extruded graphites, such blowholes are likely to be extended and aligned parallel to

the extrusion axis, appearing as stringers of elongated voids whose combined effect may be very damaging. In molded graphites they are often aligned in planar arrays normal to the molding axis. Other disparate defects encountered in less well-made graphites include: macrocracks produced by gas evolution after the formed body has become rigid, by macrostresses resulting from external restraints on contraction during baking or graphitizing, or by elastic springback when the forming pressure is released; regions of binder deficiency, such as that shown in Fig. 1, where cohesion between filler particles is almost or entirely lacking; spongy regions where, due to poor mixing, bridging, or a poor distribution of filler-particle sizes, there was an excess of binder, resulting locally in much shrinkage and much gas evolution; inclusions, usually of refractory metal carbides; voids left by vaporization of less-refractory inclusions; and "reorganized" graphite, where interaction between carbon and some impurity has produced a very highly ordered graphite structure unlike that of either the filler or the binder residue.

There are, then, two major distributions of defects within a normal polycrystalline graphite, which affect many aspects of its physical and mechanical behavior: a relatively uniform distribution of many small background defects, and a smaller number of much larger disparate defects, which may or may not be uniformly distributed. With regard to deformation and fracture, it is the interaction between the background and the disparate defects that is of particular interest.

CRACKING AND FRACTURE

Green, Zukas, and Weertman⁹ used electrostatic-discharge machining to create a relatively large artificial defect, shown in Fig. 5a, in a test bar machined from a highly oriented, hot-worked graphite. They then strained the bar incrementally in tensile creep at 2500°C, and observed its microstructure after each increment of strain. As would be expected in a brittle material, the first effect of strain was the formation of cracks, approximately normal to the tension axis, in the regions of stress concentration at the edges of the disparate defect (Fig. 5b). However, instead of propagating to fracture -- as they would normally do in a flaw-free, brittle material -- these cracks extended only for a limited distance, and then stopped. With the next increment of strain (Fig. 5c) new cracks appeared, some of them quite remote from the first, and these too grew only to rather small sizes and stopped. With each further increment of strain new generations of microcracks appeared, until (Fig. 5f) they became so numerous that they began to overlap and join. Finally one set formed a discontinuous macrocrack of critical size, and this propagated at an increasing rate to fracture.

Green, Levinson, Reissig, and Zukas⁶ observed similar behavior in less-well-ordered structures, such as that shown in Fig. 6. During tensile creep at 2500°C, microcracks formed at background defects within and between filler particles. As in Fig. 5, these microcracks were concentrated in the regions of stress concentrations at the edges of disparate defects, and the fracture paths therefore tended to extend from one disparate to the next. However, many microcracks also formed in regions remote from the disparates, and most of these did not participate in fracture. By measuring changes in distance between identifiable microstructural features, such as those indicated by letters and small arrows in Fig. 6, Green *et al.* demonstrated that measurable microstrain occurred only in material within which microcracks had formed or pre-existing voids had widened, and that all macroscopic strain of the specimen could be accounted for by the volume increase produced by this opening of cracks and voids.

Although the work of Green *et al.* was done in tensile creep at very high temperatures, the deformation and fracture behavior which they demonstrated is also typical both of much higher strain rates and of much lower temperatures. An advantage of their work was that the microcracks formed in high-temperature tensile creep are longer and more widely opened, and so more easily studied than are those formed under most other circumstances. Thus, Fig. 7 is a photomicrograph of part of a relatively tight thermal-stress crack in a premium-quality commercial graphite. This crack formed at a temperature much below 2500°C and at a very high strain rate. As would be expected, its trend is approximately normal to the principal tensile axis, and it tends to propagate from one disparate defect to the next. Between the dispartes the crack branches repeatedly, according to the local stress field and a distribution of back-ground defects which is not resolved at this magnification. Crack segments large enough to be resolved have formed elsewhere than in the main fracture path, and many microcracks have formed which could be observed only at a magnification much higher than that used in Fig. 7.

Similar cracking and fracture behavior have been reported for tests at a variety of temperatures and strain rates on a number of graphites, notably by Pears⁸ in a detailed study of the fracture behavior of ATJ-S graphite.

CONCLUSIONS

1. The sequence of events described above is believed to be typical of the deformation and fracture behavior of any polycrystalline graphite, with two exceptions: (a) The rare case of pure compression, which causes voids to close instead of open, and which will not be further considered here; (b) The more common case of a graphite specimen containing a disparate defect so large that it propagates directly to fracture at stresses so low that little or no additional cracking occurs, and there is no measurable macroscopic strain.
2. Microcracks form and open only in tension. The tension, however, may be either direct or resolved, and may be local instead of general -- produced, for example, by bending, macroscopic or microstructural buckling, or restrained thermal expansion or contraction. The cracking behavior here considered can, therefore, result not only from simple tensile loading but also from shearing, compressive, bending, or complex loads, no matter how these loads are developed. Thus, the initial failure of a polycrystalline graphite in compression is commonly by splitting parallel to the load axis, and Slagle² describes the formation of microcracks approximately parallel to the applied load in a compression test.
3. The inelastic or anelastic tensile strain that is measured macroscopically can be accounted for entirely by the dilatation produced by the opening of cracks and voids. This is a brittle behavior. However, because the microcracks involved in this behavior form first in the regions of highest stress and remain small, they have the same result as would plastic deformation in releasing strain-energy locally, transferring load to flatten the stress field, and producing a curved stress-strain diagram. So long as the accompanying dilatation is unimportant or is taken into account, this pseudo-plasticity can be treated in the same terms used for true plasticity, and it is equally useful in reducing stress concentrations at notches, providing resistance to thermal stresses, etc.
4. Similarly, the "self-stopping" behavior of microcracks and the requirement for higher stress to develop each successive generation of microcracks represent a type of pseudo-strain hardening. Strain-hardening is quite unexpected in a brittle material but has, in fact, generally been assumed in mechanical analyses of the behavior of

polycrystalline graphites.

5. The microcracks formed in a strained graphite originate at small background defects of several types, which vary in location and seriousness with differences in the filler, binder, and manufacturing procedures used to make the graphite. Necessarily, however, the background defects in a well-made graphite are as uniformly distributed in the graphite structure as are the filler particles and the interstices between them. Abnormally large (disparate) defects are important in the mechanical behavior of graphite primarily because they concentrate stresses and therefore localize the microcracks that originate at these background defects. Families of microcracks spreading out from disparates which are nearly coplanar will eventually meet and join to form a fracture path that extends from one disparate to the next. Between the disparates, the details of the fracture path are controlled by the distribution of background defects.

DISCUSSION

It has so far been explicitly assumed that the behavior of a polycrystalline graphite is perfectly brittle, involving no plasticity in any form. In fact, this is an oversimplification, although the plastic deformations that occur in such a graphite do not themselves produce macroscopically measurable strains. Thus kinking in compression, twinning in shear, and local conversion of hexagonal to rhombohedral graphite are occasionally observed even in polycrystalline graphites. Each of these structural changes must contribute an increment of microstrain, although even in sum their strain contribution is insignificantly small.

More important to an understanding of the behavior outlined above is the observation that interplanar slip undoubtedly occurs in graphite structures adjacent to opening cracks, to accommodate bowing outward of the crack surfaces. If the bending involved were purely elastic, then strain recovery would be complete when the external load producing the strain was removed. In general, macroscopic strain recovery is not complete, in tests either at room temperature or at elevated temperatures. On the micro-scale, Slagle² observed that cracks opened in tension at room temperature closed only partially when stress was relieved by fracture, and Green *et al.*⁶ also saw only partial crack closure after tensile loads were removed at 2500°C. On the other hand, Seldin¹⁰ has shown that in many cases the strain residual from a low-temperature test is completely recovered if the tested specimen is re-heat-treated to its original graphitizing temperature. This requires an inelastic deformation process -- presumed to be basal-plane slip, by dislocation motion -- which at higher temperatures is completely reversed, presumably by thermally activated back-diffusion of the dislocations. The shearing strain produced by slip would not itself be detectable as an axial dimensional change, but would facilitate the crack-opening whose result is measured macroscopically. The driving force for back-diffusion of dislocations is probably strain-energy stored elastically in material farther from the crack surface than that in which slip occurs. It is probably the progressive development of these back-stresses that is principally responsible for the cessation of growth of individual microcracks after a very limited amount of opening and extension has occurred in each. The failure of some specimens strained at room temperature and of most specimens strained at high temperatures to recover completely during a high-temperature anneal can be attributed jointly to relief of Mrozowski stresses by cracking and to stress-directed diffusion of carbon atoms.

Fracture occurs when, locally, microcracks become so numerous that they interact to form a crack of critical size, which propagates disastrously. At a given temperature and with a given distribution of defects, this can be expected to occur after a rather specific number of microcracks has formed. This indicates that, independent of the load required to create the final generation of microcracks, a definite amount of microcracking -- and so of macroscopic strain -- must occur to produce a fracture path. Evidently, then, the appropriate fracture criterion is the limiting amount of strain that a graphite can sustain before the critical crack is formed rather than the maximum stress. Evidently also, a more nearly uniform distribution of microcracks will permit more of them to form before a critical crack is created, and will increase both strain to fracture and ultimate strength. Reducing the number, size, and probability of preferred arrangements of the disparate defects will have this effect. So also will control of raw materials and manufacturing processes in such ways as to produce background defects that are small, uniformly distributed, and randomly oriented, all of which will be reflected in the microcracks formed from them.

As has been discussed by Taylor *et al.*,³ a very large amount of new surface is represented by the microcracks formed in advance of fracture and by the branching, irregular fracture path itself. The work of fracture is therefore much greater than would be predicted from the cross-sectional area of the graphite body which is loaded to failure.

It is evident that a polycrystalline graphite is very far from being an ideal Griffith solid, and that the history of crack growth in it is quite unlike the continuous, elastic process visualized in such a solid. If, however, it is desirable to identify "the Griffith cracks" in graphite, it is apparent that these must be the disparate defects discussed above, since the disparates are the effective stress concentrators that localize microcracking and so determine the fracture path. A Weibull type of statistical analysis is applicable to graphite only to the degree that the distribution of these disparate defects within it is statistically uniform. As has been noted above, preferred arrangements of the disparates -- due to peculiarities of material flow in the forming operation -- are common in molded graphites, and normal in extruded ones. A "weakest-link" concept is evidently appropriate to a discussion of the fracture of graphite, but a purely statistical prediction of the probability that the weakest link will occur in a critically stressed region is not likely to be valid.

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Fig. 1. Region of Binder Deficiency in a Manufactured Graphite. Light areas are voids, now filled by epoxy mounting resin.

Fig. 2. Binder Residue (Dark) Filling Voids Between Filler Particles. Small, rounded particles in binder residue are carbon black, added to reduce shrinkage.

Fig. 3. Defects Produced by Baking. Clear gray areas are epoxy-filled voids, including a rounded gas bubble in binder residue (spotted), gas-escape passages, and shrinkage cracks, largely at interfaces between binder residue and filler particles (striated).

Fig. 4. Epoxy-Filled Voids (White) in a Fine-Grained Molded Graphite.

Fig. 5. An Artificial Disparate Defect Machined into a Highly Oriented Graphite, and the Same Area After Five Successive Increments of Tensile Strain at 2500°C. (From Green et al., Ref. 9.)

Fig. 6. The Same Area in a Polycrystalline Graphite Before (a) and After (b) 5% Tensile Strain at 2500°C. Tensile Axis ("T. A.") is indicated by double-ended arrow. Local microstrain was determined by repeated measurements of distances between circled microstructural features. (From Green *et al.*, Ref. 5.)

Fig. 7. Part of a Thermal-Stress Crack in a Fine-Grained Graphite.

OBSERVATIONS OF FRACTURE PHENOMENA IN POLYGRAPHITES
AND THE RESULTING ARGUMENTS OF FRACTURE MODE AND CRITERIA

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(Extended Abstract)

Reconstruction of tensile fracture faces using progressively polished sections provides evidence that fracture in many polygraphites proceeds from larger void to larger void between the lamellae of aligned particles and primarily around the lamellae of nonaligned particles. This mode appears at temperatures from 70°F to about 4000°F where a single fracture plane predominates. Above 4500°F fracture seems to follow the grain boundaries and passes through the larger voids. There is evidence of particle rotation at these higher temperatures but little evidence of the bending of lamellae.

For typical graphites at up to 4000°F, crack initiation probably occurs at about 1/5 of the ultimate strength. At this stress microyield is observed in the general shape of the stress-strain curve; above about 40% ultimate, larger engineering types of offset become a feature. It seems probable that crack initiation really is the elastic fracture of weak "c" direction bonds in the coke particles and joining of the larger voids (background voids) by the development of these cracks through the lamellae of the particles at favored or statistical locations. The apparent strain aging occurs when the cracks meet nonaligned particles and propagation occurs when there is enough energy to overcome the "ab" bonds of the coke particle. Thus, the yield and ultimate strengths of a generic type of graphite are limited by its background void size and spacing, the larger particle sizes, and the bond between lamellae of the particles. This assumes a binder of some minimum nominal strength which seems typical.

Disparate voids in a graphite become threshold when their effective size exceeds the size of the statistically connective background voids or at about five times the larger void or larger particle sizes for these typical graphites.

Plane strain solutions of linear fracture mechanics seem to describe the behavior up to 4000°F when the "fracture energy" is based on c bonds for yield and ab bonds for fracture. Strengths of small structures and K_{IC} can be predicted rather well for this model and observations of the macrostructure. The local yielding required actually is elastic fracture of the particles so that cracks do not "run" but are formed by the joining of many small cracks.

These arguments lead to the conclusion that initial local strain relief and failure criteria, at least up to about 4000°F, would be equivalent tensile strain at failure. That is, there would be little synergistic influence of biaxial loadings within the normal range of yield. At high loading rates the material should be stiffer and perhaps 2 1/2 times stronger when the crack domains would not have time to form and preexisting voids and cracks would be threshold. At above 4500°F, initial local strain relief would be by equivalent tensile strain, but subsequent failure would be by shear. Viscoelasticity would prevail.

✓✓ A SIMPLIFIED APPROACH TO PREDICTING THE ONSET
OF FRACTURE IN BRITTLE MATERIALS *

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ABSTRACT

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A method of analysis is presented for predicting the initiation of fracture in brittle materials, e.g., graphites and particulated composites. It combines conventional stress-distribution analysis with a fracture criterion that indicates whether or not the stresses will cause the material to crack. Only the initiation of a crack is considered; subsequent crack propagation is disregarded. Strain energy and work are the two basic parameters of the fracture criterion. The validity of this approach was checked in tests of (1) notched beams, (2) notched tensile specimens, and (3) thin, washer-like specimens subjected to thermal gradients. Two graphites (ATJ and AUC) and several hot-pressed particulated composite materials were used. In all cases, test results and analytical predictions were in excellent agreement.

*Work performed under the auspices of the U. S. Atomic Energy Commission.

INTRODUCTION

The phenomenon of brittle fracture has eluded precise characterization in all attempts to predict its occurrence with a reasonable degree of accuracy. Excessive data scatter in test results discouraged attempts to arrive at a unique quantitative solution of the fracture phenomenon and led instead to statistical approaches. Most analytical methods produce very conservative results because they lack a good criterion of fracture and try instead to consider a wide range of material properties to account for wide scatter of the test data. Consequently, optimization efforts in the design of hardware are very unsatisfactory.

The fracture of a brittle material occurs in two distinct phases: (1) fracture initiation and, (2) fracture propagation. For example, a thin, washer-like specimen heated on its o.d. and simultaneously cooled on its i.d., will develop a radial temperature gradient that will eventually cause the formation of a small (microscopic) radial crack at the specimen i.d. (Fig. 1). This formation of the first small crack is termed fracture initiation. At higher gradients, this crack will grow towards the o.d. while at least one more crack forms and begins to grow before a crack reaches the o.d. This subsequent crack growth and crack formation is termed the fracture-propagation phase and is not discussed further. The method of analysis presented herein pertains to fracture initiation only.

Four general areas are discussed: (1) basic assumptions, (2) derivation of a criterion of fracture initiation, (3) application of the method, and (4) experimental verification.

BASIC ASSUMPTIONS

Macroscopic observations of many fracture tests of mostly reactor-grade graphites indicated the following similarities:

- Initial fracture could always be attributed to a tensile rather than a compressive or a shear field.
- Fracture occurred abruptly, without any apparent local deformation.
- In bending tests, fracture occurred in a series of short cracks of finite length rather than as a continuously growing crack.
- Test-data scatter was minimal providing the specimens were (1) carefully selected as to uniformity and, (2) tested in such a way as to restrict deflections to the primary loading direction.

The observations plus certain theoretical considerations led to the following basic assumptions:

- Initial fracture is due to the tensile stresses acting normal to the fracture-surface area.

- The material behaves elastically.
- Initial fracture occurs when the average tensile strain energy calculated in terms of the tensile stresses normal to the fracture surface equals the average energy (or work) required to cause material separation as determined in a tensile test.
- In tension, the initial fracture area is the entire cross-section.
- In bending, the initial fracture area is only a portion of the cross-section as determined by both the shape of the strain-energy curve and the material characteristics. It is further assumed that the shape effect can be simply expressed as a linear function of the location of the centroid of the tensile portion of the strain-energy curve, i.e., $A_I = n\bar{A}$, where A_I is the initial fracture area, $\bar{A}$ is the area between the fracture origin and the centroid of the curve, and n is an empirically determined factor.

A criterion of fracture initiation is derived from these assumptions by using strain energy applicable to a uniaxial stress field.

DERIVATION OF A CRITERION OF INITIAL FRACTURE

An expression for the strain energy normal to area dA is obtained from the conventional expression for the strain-energy dU (Ref. 1) by assuming a unit length in the direction of the stress.

$$dU = \frac{1}{2} \sigma \epsilon \, dA$$

where

U = strain energy

σ = stress

ϵ = strain

A = area.

Because stress is proportional to strain, ϵ can be replaced by σ/E and the total strain energy is:

$$U = \frac{1}{2} \int \frac{\sigma^2}{E} \, dA. \quad (1)$$

Defining the limits of the integration as the initial fracture area and assuming that E is constant in this location, the general formula for the total strain energy becomes:

$$U = \frac{1}{2E} \int^I \sigma^2 \, dA. \quad (2)$$

The average strain energy is obtained by dividing Eq. (2) by the initial fracture area:

$$\frac{U}{A} = \frac{\frac{1}{2E} \int^I \sigma^2 dA}{\int^I dA} \quad (3)$$

The criterion for fracture initiation is then established by equating the average strain energy for the anticipated initial fracture surface (Eq. 3) and the average strain energy required to cause material separation (determined by a tensile test):

$$\frac{\frac{1}{2E} \int^I \sigma^2 dA}{\int^I dA} = \frac{\frac{1}{2E} \int^I \sigma_U^2 dA}{\int^I dA}$$

where

σ_U = ultimate tensile stress (a constant).

This equation can be simplified to the following general formula for fracture initiation

$$\left(\frac{\int^I \sigma^2 dA}{\int^I dA} \right)^{1/2} = \sigma_U \quad (4)$$

APPLICATION

There are three parameters in Eq. (4): initial fracture area, stresses (normal to this area), and ultimate tensile stress. If the stresses are expressed as a linear function of a load, P , then Eq. (4) can be solved for P , the fracture-initiation load. Equation (4) can also be used for finding the size of the initial fracture area if the stresses can be written as an integrable function. The application of Eq. (4) to practical problems will be illustrated on several examples.

The stress distribution in the initial fracture area must account for any large concentration of strain energy, e.g., energy associated with a notch. A stress-analysis technique based on the concept of finite elements is used at IASL in a digital computer program called ARBAS to find these stresses. If the stresses are due to temperature gradients, the temperatures needed as input to ARBAS must be calculated first.

Consider a round, annular, notched tensile specimen whose initial fracture area is the entire cross-section through the root of the notch. The stresses are computed from the ARBAS code for a unit axial load, i.e., $P = 1$, so that the final stresses are equal to $P \sigma_r$. Substituting $P \sigma_r$ for σ and

$2\pi \int_{r_i}^{r_o} r dr$ for $\int^I dA$ into Eq. (4) and simplifying,

$$P \left(\frac{2 \int_{r_i}^{r_o} \sigma_r^2 r dr}{r_o^2 - r_i^2} \right)^{1/2} = \sigma_U \quad (5)$$

Equation (5) can also be solved for the fracture-initiation load, P,

$$P = \frac{\sigma_U}{\left(\frac{2 \int_{r_i}^{r_o} \sigma_r^2 r dr}{r_o^2 - r_i^2} \right)^{1/2}} \quad (6)$$

For a simply loaded beam of constant width the formula for determining the size of the initial fracture area in bending has been given above as

$$A_I = n \bar{A} \quad (7)$$

Solving Eq. (7) for n,

$$n = \frac{A_I}{\bar{A}} \quad (8)$$

For an unnotched (solid) beam, the stresses are given by the familiar formula

$$\sigma = \frac{My}{I} \quad (9)$$

where

M = bending moment

y = distance from the neutral axis

I = moment of inertia.

Both A_I and $\bar{A}$ can be calculated from this stress function. The centroid of the strain-energy curve is at one eighth the beam depth from the extreme fiber. Therefore, $\bar{A} = (d/8)w$, where d is the beam depth and w, width. The initial fracture area, A_I , is calculated from Eq. (4) by substituting the stress function and integrating over the limits of $m(d/2)$ to $d/2$, where m is an unknown factor that defines the boundary of the fracture area. The initial fracture area, then, is $(d/2)(1 - m)$. Equation (4) thus becomes

$$\left(\frac{M^2/I^2 \int_{m d/2}^{d/2} y^2 dy}{d/2 (1-m)} \right)^{1/2} = \sigma_U \quad (10)$$

Solving Eq. (10) for m,

$$m = \frac{-1 + \left[1 - 4 \left(1 - \frac{12}{d^2 K^2} \right) \right]^{1/2}}{2} \quad (11)$$

where

$$K = M/I.$$

And, finally,

$$n = 4(1 - m). \quad (12)$$

The initial fracture area can now be found from Eq. (7) for any beam geometry because $\bar{A}$ can be obtained by a semigraphical technique. Equation (4) can be solved for either the initial fracture load, P , or for the ultimate tensile stress, σ_u , as explained in the first example.

The integration in Eq. (4) is actually performed as a summation because it can be based directly on the ARBAS output, providing the grid used in the mathematical model is fine enough to give a good approximation of the exact integral.

VERIFICATION OF METHOD

The validity of this approach for predicting the onset of fracture has been demonstrated experimentally by the three tests described below.

Case 1 - Sharply Notched, Solid, Round Tensile Specimen

Two round tensile specimens, one unnotched and the other sharply notched, made from 46-vol % NbC-TaC-C composite material were tested to determine the ultimate tensile stress and to verify fracture initiation, respectively.

The load was applied by an Instron testing machine. The specimens (Fig. 2) were held in position by a self-aligning universal joint at one end and by a cable at the other. The unnotched specimen was tested first and broke at a load of 760 lb, equal to an ultimate tensile stress of 7720 psi.

The notched specimen was analyzed with the ARBAS code to determine the stress distribution through the critical cross-section at the root of the notch. This information was used in Eq. (6) to find the fracture-initiation load. The calculated load was 522 lb. This compared very well with the test load of 512 lb.

Note that the theoretical tensile stress at the root of the notch exceeded 80,000 psi at failure (Fig. 3) and that the load-carrying capacity of this specimen was reduced by only 31%.

Case 2 - Sharply Notched, Constant-Width Beams

All specimens in this test were made from the same billet of ATJ graphite and had the same orientation.

The test apparatus permitted only one degree of freedom to ensure a beam deflection that was compatible with the assumptions of a simply loaded beam, $\sigma = My/I$ (Fig. 4). Each specimen was machined for a close slip-fit between the parallel steel plates. The load was applied at the third points by the smaller pin-guided steel plate at the top of the fixture. Twenty specimens of each type of notched beam were tested to investigate scatter characteristics.

The size of the initial fracture area for the notched beams was determined from the test result for an unnotched beam of the same material as discussed in the preceding section. The empirical factor, n , was found to be 2.5. An ultimate tensile stress of 3600 psi was obtained as the average of 22 individual tests.

The load applied to a beam is highest at the moment of fracture initiation. Consequently, the maximum test load was compared to the predicted fracture-initiation load per Eq. (4). Test results and analytical predictions were in excellent agreement as shown in Table 1.

Case 3 - Steady-State, Thermal-Stress Fracture of Thin, Annular Graphite Disks

All test specimens were made from the same AUC graphite extrusion. Thin, annular disks were fractured by thermal stress in a test jig by creating a radial temperature gradient across the specimen. The analytical solution involved temperature dependencies of the material properties as well as the empirical evaluation of mechanical and thermal boundary conditions. The analysis showed that the value calculated from Eq. (4) was 2400 psi as compared to the ultimate tensile stress of 2200 psi for this material. Because the analytically determined value exceeded the ultimate tensile stress, the initiation of a fracture was confirmed. A complete description of the test and analysis is presented in Ref. 2.

ACKNOWLEDGMENTS

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Table 1. Analysis vs. Test for Notched Beams^a

Identification	Initial Crack Length, in.	Test Load, lb	Predicted Load, lb	Difference, %	No. of Specimens Tested	One Standard Deviation, lb
Notch Depth, 0.05 in.	0.059	32.7	36.8	+ 12.5	20	2.1
Notch Depth, 0.15 in.	0.032	19.2	17.1	- 10.9	20	1.5
Notch Depth, 0.25 in.	0.023	10.6	8.9	- 16.0	20	0.8

^aBasic beam dimensions, 0.50 in. deep by 0.25 in. wide by 5 in. long (4 in. between supports). The notch is at the center line of the beam, in the lower portion, i.e., in tension.

STEADY-STATE, THERMAL-STRESS TEST

Specimen Size - 1.200 x 0.500 x 0.0625 in.

Material - 50 v/o ZrC-C Composite

Fracture Initiation

Same Specimen After Exposure to Higher
Temperature Gradient Showing Propagation

Fig. 1. Fracture Initiation and Propagation.

UN-NOTCHED SPECIMEN

PHOTO

ALL OTHER DIMENSIONS
SAME AS ABOVE

NOTCHED SPECIMEN

PHOTO

Fig. 2. Case 1 - Tensile Specimens.

Fig. 3. Theoretical Tensile Stress Distribution Through Notch.

Fig. 4. Restrained Beam Test Fixture.

THE MECHANICAL PROPERTIES OF GRAPHITES
AND THEIR RELATION TO A PHYSICALLY BASED
STATISTICAL THEORY OF FRACTURE

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INTRODUCTION

Graphite is a large volume industrial material of great importance in high temperature technology. In bulk form, it is characterized by weakness, low modulus, significant porosity, susceptibility to preferred orientation effects, and a statistical (rather than well defined) brittle fracture strength. In fiber form, it can achieve great strength and modulus as well as low porosity.

There are a great number of bulk graphites and fibers currently available which span a tremendous range of properties. The intent of this note is to develop a simple single scheme from which the majority of these properties may be rationally understood. Specifically, the topics of statistics of fracture, grain size effects, open and closed porosity effects, preferred orientation effects, and bi-axial failure conditions will eventually be covered by this scheme. These concepts can logically be expected to be related as they should all be affected by the same fundamental flaw, or inherent characteristic, which limits the mechanical properties of graphite. It is the purpose of this note to point out a logical set of candidates for this fundamental flaw, which behave similarly, and to demonstrate the value of these concepts in correlating the mechanical properties of various forms of graphite.

BACKGROUND

The logical areas in which to seek the inherent characteristic(s) which inhibit the mechanical properties of graphite are the binder phase and the graphite grains or particles which make up the particular polygraphite* under consideration. The effects of porosity, while real, will not form a sufficient basis for the fundamental understanding of the mechanical properties of pore-free polygraphites (dense pyrolytic graphite, fibers, or completely reimpregnated bulk polygraphites). Therefore, porosity effects will be considered later as a modification of the properties of pore-free polygraphite.

Taking into account the high mechanical and structural anisotropy of graphite crystals (strong covalent bonding in plane, weak vander walls bonding out of plane), and the nearly amorphous nature of much of the binder, one finds that a composite ceramic equivalent of pore-free polygraphites is a low modulus glass containing a high volume fraction of included mica crystals. In this system, as well as in the polygraphite system, the questions of importance are the relative strength of the matrix, the matrix-particle bond, and the weakest strength of the included crystals.

*The term polygraphites is used to denote all graphites other than essentially perfect graphite crystals.

The strength of the carbon binder in a pore-free polygraphite is difficult to determine. Glassy carbon, for example, at room temperature (density of less than 1.5 g/cc³) exhibits a tensile strength of 6 ksi as measured with specimens containing nicks and chips in the gauge section (Ref. 1). On the other hand, isotropic pyrolytic carbons (densities in the range 1.5 g/cc to 2.0 g/cc) exhibit bend strengths in the range of 30 to 40 ksi and laminar anisotropic pyrolytic carbons (densities in the range of 1.5 g/cc to 2.16 g/cc) exhibit bend strengths in the range of 37 to 70 ksi (Ref. 2). Carrying this further, carbon fibers have been reported to have tensile strengths considerably in excess of 100 ksi. It is therefore difficult to conclusively argue that the matrix of a pore-free polygraphite would be the weakest link in the polygraphite.

The graphite grain, or more accurately the crystal, is highly anisotropic. For example, assuming as deposited pyrolytic graphite represents an approximation to the graphite grain or crystal, the tensile strength in the basal plane is approximately 14 ksi while the strength perpendicular to the basal plane is approximately 1 ksi (Ref. 3)*. The room temperature shear or torsional strength on the basal plane is in the range of 1 to 2.5 ksi (Ref. 4 and 5)*. This material may be hot extruded so as to increase crystal perfection raising the basal plane modulus from 3.2×10^6 psi to 82×10^6 psi (Ref. 6), very similar to graphite fiber data. Natural graphite crystals are ordinarily cleaved into thin sections for transmission electron microscopy by stripping with adhesive tape. These data, coupled with the presumed weak Van der Waals bonding in the "c" direction, indicate that the basal plane bonding is a logical candidate for an intrinsic source of weakness under local tension or shear in pore-free polygraphites.

An anisotropy of the graphite crystal poses a second logical reason for the basal plane bonding as an intrinsic weakness. The low (sometimes negative) in plane expansion coefficient coupled with a high modulus and the high out of plane expansion imply that on cooling a polygraphite from the high graphitization or fabrication temperature, high residual tensile strain (and stress) fields are developed in the "c" directions of the graphite crystals. (Ref. 7). These strains could either lead to easy fracture of the "c" bonding and crack formation under applied load or to the preexistence of basal plane cracks on the order of the grain diameter. This also could impose an intrinsic limit to the strength of pore-free polygraphites.

The third logical candidate as a source of intrinsic weakness is the matrix - graphite grain bonding. It is easy to visualize a carbon matrix bonding strongly to the "a" faces, or ends of the basal planes, as there are a large number of carbon atoms which are available to form some type of covalent bonds. The bonding to the "c" faces is more difficult to envision as being strong. The atoms in the "c" planes have "used up" their covalent bonding, by and large, in forming hybrid covalent bonds with adjacent basal plane carbon atoms and thus appear to be not immediately available for strong out of plane bonding. Therefore, this matrix - graphite grain bond is a logical candidate as an intrinsic weakness for limiting the mechanical properties of pore-free polygraphites. In fact, due to thermal expansion differences, at room temperature this bond could have already degraded into a preexisting crack on the order of a grain diameter, or would do so very readily under local tensile or shear stress.

*Significantly lower values have been reported in "Some Thermophysical Properties of Pyrolytic Graphite to 5000°F" (1961), a summary report to Lockheed Missiles and Space Company by Southern Research Institute 6216-1209 - XVII.

If we disregard the matrix as the weak link or critical flaw in pore-free polygraphite, the logical candidates for this critical flaw are the "c" axis bonding, the adhesion of the matrix to the "c" plane, and the thermal expansion differences and how they affect the "c" axis bond and matrix - grain bond. The common factor here is the relationship to the "c" axis and the fact that many elemental flaws on the order of the grain diameter must be expected. This sort of critical flaw also, of course, leads naturally into a physical set of reasons through which one might expect to relate the anisotropy of the mechanical properties to the anisotropy of the grain orientation distribution.

The Model

As outlined in the previous section, the critical flaw for non-porous polygraphites has been argued as having a relation to the "c" axis resulting in either a preexisting crack parallel to the basal plane or one which can be readily developed under either local tension or shear. We reinforce this supposed "c" axis effect by quoting from Smith (Ref. 8) as follows.

"Below 2500°C, the fracture history of H4LM poly graphite in the tensile tests seems to be that originally described by Jenkins (9) for another reactor [poly] graphite: appearance, with increasing strain, of a multitude of fine, independent, interlamellar cracks within the graphitized coke grains; growth of these to some small but microscopically observable size, after which growth apparently ceases; finally, when they become very numerous, joining of the most favorably oriented of these cracks to form a fracture path... So long as fracture is by the low temperature mechanism, apparent ductility of the [poly] graphite is low, and permanent extension in advance of fracture is not more than a few tenths of one percent (9)".

(a) Description of Favorably Oriented Single Flaws

For calculation purposes, it is not convenient to attempt to describe the development of such flaws; their number, location and stress at which they occur. We, instead, replace this development of the flaws by assuming that since each favorably oriented grain or matrix-grain boundary has the potential to develop into a flaw of approximately one grain in extent, we may therefore assume that all favorably oriented grains have developed such a flaw. Favorably oriented, in the case of tensile loading is defined as a grain having an orientation such that the local resolved tensile stress appearing across the "c" plane (or matrix - grain interface) is 80% (or some fixed fraction λ) of the maximum average value occurring in the sample (more rigorous ways to avoid introducing this somewhat arbitrary parameter, 80% or λ , will be discussed in a later publication).

Assume that, relative to the principle loading axis, the "c" direction can be described by some probability of orientation function, $P(\varphi, \theta)$ as illustrated in Fig. 1. The resolved normal tensile stress, σ_n , is given in terms of the applied stress, σ , by

$$\sigma_n = \sigma \cos^2 \theta \quad (1)$$

Therefore the fraction of favorably oriented grains, f_λ , in which

$$\sigma_n / \sigma \geq \lambda \approx .80 \quad (2)$$

are found from the probability function, $P(\varphi, \theta)$ by

$$f_{\lambda} = 2 \int_{0=0}^{2\pi} \int_{0=0}^{\cos^{-1}(\sqrt{\lambda})} P(\varphi, \theta) d\theta d\varphi \quad (3)$$

For randomly oriented grains* (and isotropic pore-free polygraphite),

$$P(\varphi, \theta) = \frac{\sin \varphi}{4\pi} \quad (4)$$

and hence

$$f_{\lambda} = 1 - \sqrt{\lambda} \quad (5)$$

For $\lambda = .8$, $f_{\lambda} = .105$ indicating that approximately ten percent of all grains should be considered as somehow flawed just prior to failure.

(b) "Coincidence Alignments" Forming Larger Flaws

These flaws, if statistically closely spaced in some segment of the specimen, are capable of acting as a single Griffith flaw of significantly larger size through a local "coincidence alignment". To develop this concept, consider modeling the grains as small cubes of size, g , stacked in layers as in Figure 2. Assume, of course, that the binder or matrix is present as a sufficiently thin layer over the surface of the cubical grains so that we may ignore considering it further.

Pick, at random, a grain which satisfies the definition of favorably oriented. In the plane, this grain has four neighbors which share faces and four which share edges. Select one of the neighbors. The probability that it is favorably oriented is f_{λ} . The probability that only one neighbor out of the eight neighbors is favorably oriented is given by $8 f_{\lambda} (1 - f_{\lambda})^7 \approx .385$. The probability (P_{m8}) of m of the eight neighbors being locally aligned is

$$P_{m8} = \frac{8!}{m! (8-m)!} f_{\lambda}^m (1 - f_{\lambda})^{8-m} \quad (6)$$

For the hypothetical value, $f_{\lambda} = .105$, the probability that all eight neighbors to a favorably oriented grain are also favorably oriented is thus $P_{88} \approx 5 \times 10^{-8}$. The probability that not all of the neighbors are similarly aligned is $1 - P_{88}$. In a given volume, V , of material there are N grains, where

$$N = V/g^3 \quad (7)$$

Pick one of the grains at random. The probability that it is not favorably aligned is $1 - f_{\lambda}$. The probability that it is favorably aligned but does not have eight favorably aligned neighbors is $f_{\lambda} (1 - P_{88})$. Thus, the probability that a grain chosen at random is neither favorably aligned nor if it is favorably aligned it does not have eight favorably aligned neighbors is given by $(1 - f_{\lambda} P_{88})$. The probability that none of the N grains in a volume V consists of a favorably oriented grain surrounded by eight favorably oriented neighbors is thus $(1 - f_{\lambda} P_{88})^N$. Therefore, the probability, $P_{88}(V)$, of finding in a volume, V , a favorably oriented grain surrounded by eight favorably neighbors is

$$P_{88}(V) = 1 - (1 - f_{\lambda} P_{88})^N \quad (8)$$

Taking, as an example, $V = 1$, $g = 10^{-3}$, $f_{\lambda} = .05$ we find that the probability of finding a coincidence alignment of nine grains arranged in a square is 0.995,

*Note that grain anisotropy is a fundamental consideration

which is to say it is a near statistical certainty that nine small cracks will in essence join up to produce a significantly larger flaw. Such a large flaw will further reduce the calculated strength relative to considering merely a single flaw, and should be incorporated into theoretical considerations of the strength of graphite. This basically describes the development of the concept of "coincidence alignment".

Suppose that we pick n contiguous grains beginning from one grain which satisfies the favorable orientation criterion. Then, the probability, P_{mn} , that set of n contiguous grains contains m grains also favorably oriented is

$$P_{mn} = \frac{n!}{m! (n-m)!} f_{\lambda}^m (1 - f_{\lambda})^{n-m} \quad (9)$$

By similar reasoning, the probability, $P_{mn}(V)$ of finding an array of m grains satisfying the favorably oriented condition out of a contiguous set of n grains in a specimen of volume V is thus

$$P_{mn}(V) = 1 - (1 - f_{\lambda} P_{mn})^{V/g^3} \quad (10)$$

Now, it seems reasonable that if in a collection of n contiguous grains, the fraction of them m/n , which are favorably aligned is high, say $m/n = h \approx 0.9$, then we may effectively consider this entire arrangement to behave as if it were entirely favorably aligned and could thus act as a flaw composed of n contiguous grains (the arbitrary parameter h can be eventually eliminated as will be discussed in a later publication. Thus $P_{mn}(V)$ with $m \approx hn$ represents the approximate probability of having n contiguous grains in a particular planar pattern all acting as if they were similarly aligned.

(c) Shape of the "Coincidence Alignment"

While the "coincidence alignment", or flaw, can be of any shape which satisfies the restriction of n contiguous grains, it is possible to consider only a limited number of arrangements. The reader familiar with fracture mechanics (Refs. 10 and 12) will recognize that the circular penny shaped crack of a given area is more detrimental to the strength of a material than any elliptical planar flaw, of the same area regardless of eccentricity, and is therefore lead to postulate that most of the fracture behavior of poly-graphites can probably be obtained by assuming that the coincidence alignments form circular crack-like planar domains*. The physical picture is given in Figure 3.

Polygraphites, as virtually any other material which can be made to fail in a brittle manner, possess a fracture toughness parameter (Ref. 13), K_{IC} . This fracture toughness contains the true fracture energy and is usually obtained by direct experiment. The tensile failure criterion for a circular crack of radius according to Reference 11 is

$$\sigma = K_{IC} \sqrt{\pi/2^a} \quad (11)$$

A coincidence alignment of n grains in extent has a radius approximately given by

$$a = 1/2 g \sqrt{N/\pi} \quad (12)$$

*Exceptions to this generalization will be given in a later paper. Their preliminary influence is to widen the failure histograms slightly.

(d) Uniaxial Tensile Fracture Due to Crack Distributions

Suppose a material contains a crack size distribution, say $(1 - s(a, V))$ where $[1 - s(a, V)]$ represents the probability of finding within a volume V at least one crack of size a ; what is the description of failure? For a specimen to fail under the stress σ , we must have at least one crack whose size is greater than

$$a = (\sigma / K_{IC})^2 2/\pi \quad (13)$$

For a specimen not to fail under stress, we must not have a crack of size greater than that given by equation (13).

The probability of not having a flaw composed of $\tilde{n}$ contiguous grains (of flaw radius $a = \sqrt{n/\pi}$) is $s(\tilde{n}, V)$ and is

$$s(\tilde{n}, V) = 1 - P_{m\tilde{n}}^{\sim}(V) \quad (14)$$

The probability, $S(n, V)$ of not having a flaw larger than n grains in extent is thus

$$S(n, V) \cong \sum_{\tilde{n}=n}^{\infty} S(\tilde{n}, V) \quad (15)$$

The probability, $S(a, V)$ is thus

$$S(a, V) = S(n, V) \quad (16)$$

which is equal to the probability of the specimen not failing under a stress given by

$$\sigma = K_{IC} \sqrt{\pi/2a} = K_{IC} \sqrt{\pi/g} / (n/\pi)^{1/4}$$

Fracture of polygraphites is thus inherently statistical. Thus, we may write for the probability of no failure

$$P(\sigma = K_{IC} \sqrt{\pi/g} / (n/\pi)^{1/4}) = \sum_{n=n}^{\infty} (1 - P_{m\tilde{n}}^{\sim}(V)) \quad (17)$$

Another way of expressing the same thought is that the probability that a specimen will not fail due to a "coincidence alignment" n grains in extent is

$$P_{(n)} = \sum_{\tilde{n}=n}^{\infty} (1 - P_{m\tilde{n}}^{\sim}(V)) \quad (18)$$

where $P_{m\tilde{n}}^{\sim}(V)$ is given by equation (10) with the parameters m and n fixed as discussed previously.

Uniaxial Tension Results

The probabilities of no failure as a function of size of "coincidence alignment" for various specimen volumes and grain sizes as well as λ values are given in Figures 4 to 6. As discussed in the appendix, a reasonable range of λ values can also be interpreted as an anisotropy in the grain orientation

*This approximation can be shown to be sufficiently accurate for the particular use intended here.

distribution, and Figures 4 to 6 imply that the theory forms a basis for understanding a substantial portion of the anisotropy of polygraphites.

The stresses implied by the previous figures are shown in Figures 7 to 9 in which the measured K_{IC} fracture toughness value, 1.4 ksi/in, for POCO polygraphite (Ref. 13) has been used to convert size of coincidence alignment to strength. POCO is reported in the manufacturers literature as having a grain size of "less than 0.001 inches" and thus Figure 9 with $\lambda = .8$ should be compared to the reported tensile strength of approximately 10 ksi (Ref. 14). The agreement is good considering that the theory assumes no porosity and POCO is porous. Porosity effects will be discussed later. If one did not use the "coincidence alignment" concept, and merely checked the reasonableness of a Griffith mechanism based upon the measured grain size and fracture energy, one would be led to the conclusion that Jenkins (Ref. 15) arrived at, i. e., the fracture of polygraphite implies either a Griffith flaw many times larger than the grain size or an additional stress intensification mechanism perhaps based upon a dislocation pile-up.

If one were to attempt to correlate the present approach with the bulk anisotropy of polygraphites, a critical step is the question of the anisotropy of the fracture toughness, K_{IC} . The fracture toughness, K_{IC} , has been measured both parallel and perpendicular to grain in ATJ polygraphite (Ref. 16). The fracture toughnesses parallel to grain and perpendicular to grain are approximately 0.8 and 0.6 ksi in. This implies that the grain orientation effect on the formation of "coincidence alignments" must also be augmented by the effect of grain orientation on fracture toughness. Nevertheless, the theoretical concept of "coincidence alignments" is important in understanding the anisotropic graphite failure behavior.

The effects of specimen volume, according to the present theory, are shown in Fig. 10 in terms of percentage of small volume strength. Data on ATJ, from Ref. 17, are also shown as well as Weibull (Ref. 18) predictions for the volume effect on strength. It is seen that the present theory agrees well with experiment, while the volume effect is not of the Weibull form.

The effect of grain size on polygraphite strength can only be simulated if the toughness, K_{IC} is known. Assuming a grain size independent toughness, the grain size effect will be an exact inverse square root dependence if the ratio of grain size to specimen size is held constant, Fig. 11. Data from Ref. 19 on various polygraphites with significant porosity (densities approximately 1.6 to 1.9) with unknown toughnesses and specimen to grain size ratios appear to cluster around the theoretical line. This grain size strength relationship has also been previously discussed (Ref. 20) as well as porosity effects which will be considered in another paper.

CONCLUSION

In summary, when applied to the tensile properties of a non-porous polygraphite, the present theory appears to give fairly accurate estimates of strength, specimen effects, the effect of grain size and anisotropic grain orientation, and to be a direct physically based statistical theory of fracture. This theory is being extended to anisotropic materials, porous materials, and multiaxial stress states. In a more detailed version, the stress-strain properties will also be considered.

APPENDIX I THE ELIMINATION OF ADJUSTABLE PARAMETERS

The adjustable parameter, λ , was introduced to provide a convenient means for identifying which grains of a polygraphite must be considered as capable of acting as a flaw or crack. The parameter h was introduced to define a large region of the polygraphite which through a "coincidence alignment" of most of its grains, given by the fraction h , could be considered as acting as a large flaw. Both of these parameters can be eliminated by simulating polygraphite in a finite element computer analysis and examining the similarity of the stress fields in the vicinity of a "coincidence alignment" to an ideal large Griffith flaw.

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Fig. 1. Description of c Direction by a Probability of Orientation Function, $P(\phi, \theta)$.

Fig. 2. Model of Polygraphite as Randomly Oriented Cubic Grains Stacked in Layers (Binder or Matrix Assumed Thin Enough To Be Ignored).

Fig. 3. Model of Circular "Coincidence Alignment," or Flaw.

Fig. 4. Probability of No Failure as a Function of Size of "Coincidence Alignment" for $V = 1 \text{ in.}^3$, $g = 0.002 \text{ in.}$, and $f_\lambda = 0.05$ to 0.2 .

Fig. 5. Probability of No Failure as a Function of Size of "Coincidence Alignment" for $V = 0.01 \text{ in.}^3$, $g = 0.002 \text{ in.}$, and $f_\lambda = 0.05$ to 0.2 .

Fig. 6. Probability of No Failure as a Function of Size of "Coincidence Alignment" for $V = 1 \text{ in.}^3$, $g = 0.0001$ to 0.1 in. , and $f_\lambda = 0.1$.

Fig. 7. Probability of Survival as a Function of Stress for $V = 0.1 \text{ in.}^3$, $g = 0.002 \text{ in.}$, and $f_\lambda = 0.05 \text{ to } 0.2$.

Fig. 8. Probability of Survival as a Function of Stress for $V = 1 \text{ in.}^3$, $g = 0.002 \text{ in.}$, and $f_\lambda = 0.05 \text{ to } 0.2$.

Fig. 9. Probability of Survival as a Function of Stress for $V = 1 \text{ in.}^3$, $g = 0.0001 \text{ to } 0.1 \text{ in.}$, and $f_\lambda = 0.1$.

Fig. 10. Effect of Specimen Volume on Relative Strength.

Fig. 11. Effect of Grain Size on Strength of Polygraphites.

BIAXIAL MECHANICAL PROPERTIES OF AXF-5Q GRAPHITE TO 4000°F

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ABSTRACT

Fracture strengths and strains of AXF-5Q, a fine-grained isotropic polycrystalline graphite, have been measured in several biaxial stress states at 70°F, 3000°F, and 4000°F. Hollow cylindrical specimens of one-inch inner diameter, 0.050-inch wall thickness, and one-inch gage length were simultaneously pressurized internally and loaded axially to obtain, with different ratios of load to pressure, a variety of stress states covering the tension-tension and compression-tension quadrants of the biaxial stress plane. Supplementary uniaxial tensile and compressive data were obtained on solid cylindrical specimens.

The fracture data show that the maximum tensile stress at failure is decreased below the uniaxial tensile strength in the tension-tension quadrant, is equal to the uniaxial tensile strength in the compression-tension quadrant for applied compressive stresses which are less than the applied tensile stress, and decreases towards zero as the compressive stress approaches the uniaxial compressive strength. Biaxial strength increases with increasing temperature in approximately the same ratio as does the uniaxial tensile strength. The strain data shows that, as might be expected from consideration of Poisson's effect, the maximum tensile strain at failure is smaller in biaxial tension, and greater in compression-tension, than in uniaxial tension.

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INTRODUCTION

The work described here was aimed at characterizing the mechanical behavior of AXF-5Q graphite under biaxial states of stress covering the tension-tension and compression-tension quadrants of the stress plane. Biaxial tests were performed at 70°F, 3000°F, and 4000°F. The data presented includes the stresses and strains measured at fracture. The biaxial specimens were hollow cylinders subjected to internal pressure and axial load. The test techniques used represent the results of an effort to minimize load misalignments, stress concentrations due to load introduction, and penetration of the pressurizing medium into the specimen.

Prior work in biaxial testing of bulk graphites provides some data on ATJ and ATJ-S graphites at room temperature (References 1 and 2) and on Graphitite "G" at room temperature and 2000°F (Reference 3). However, considering the importance that failure criteria under multiaxial loading have to the designer of all but the simplest structural components, it is surprising how little biaxial failure data there is. The study reported here is believed the first to include biaxial data at temperatures above 2000°F. The number of specimens tested at elevated temperature was limited by economic considerations to one, two or three per biaxial stress state. However,

at room temperature, eight specimens were tested in each stress state to lend some statistical significance to the data. For development of multiaxial failure criteria, the grade of graphite studied (AXF-5Q) provides a particularly simple case because it is mechanically isotropic; at the same time AXF-5Q is of interest, structurally, because of its relatively high strength.

MATERIAL AND SPECIMENS

Test specimens were taken from forty-four plates, approximately two inches thick by four inches wide and twelve inches long, of AXF-5Q graphite supplied by the Poco Graphite Company. Figure 1 shows the distribution of tensile strengths obtained by testing several (three or four) uniaxial tensile specimens from each plate. The scatter in properties is fairly large, as might be expected for a brittle material, and there was significant variation in the average tensile strengths of the plates. The biaxial specimens were allocated to each biaxial test condition in a deliberately "random" manner in an attempt to minimize the effect of parent plate variability. That is, the several specimens tested in a single stress state were taken from different plates so that "weak", "average", and "strong" parent plates were represented in the same proportion as in other stress states. Before excising the test specimens, the plates were nondestructively inspected using ultrasonic pulse-echo scanning. Areas of the plates which exhibited significant flaw indications were avoided in the preparation of specimens.

Figure 2 shows examples of the uniaxial tension, biaxial, and uniaxial compression specimens tested. The tensile and biaxial specimens are 4 inches long; the compressive specimen is 2.5 inches long. Tolerances relating to concentricity and perpendicularity of the gage section to the loading surfaces were maintained in the 0.0005 inch range as indicated in Figure 3 for the biaxial specimen. In addition to the specimens shown in Figure 2, some ring specimens (1.00 inch inner diameter, 0.050 inch wall thickness, and 1.00 inch long) were tested in hoop tension.

TEST TECHNIQUES

The biaxial specimens and the uniaxial tensile and compressive specimens were tested in "rigid-fixtures" adapted to Instron test machines. The rigid fixture, described in Reference 4, comprises a set of guide bushings mounted in a heavy steel yoke; the rods which load the specimen are carefully matched dimensionally to each bushing so that nonaxial and rotational motions are limited by the small (.0005 inch) rod-to-bushing clearance.

A typical setup is shown in Figure 4. Bending strains measured in uniaxial loading of tensile and compressive specimens were less than two percent of the average axial strain at fracture; bending strains measured on biaxial specimens amounted to less than three percent of the average axial strain at failure. Friction between load rods and guide bushings was measured to be less than two percent of the axial load.

The hoop tension ring specimens were tested under internal pressure without axial load in a fixture patterned after that of Reference 10.

Figure 5 shows the biaxial specimen assembly for testing at room temperature. A thin rubber balloon is used to prevent penetration of the pressurizing fluid (water) into the graphite. This precaution was deemed necessary because the graphite is porous and would be expected to behave differently if the pores were filled with high pressure fluid. Strains were measured in the axial and circumferential directions using bonded strain gages and mechanical extensometers attached to the outer diameters of the specimen.

Figure 6 shows the biaxial specimen assembly for testing at elevated temperatures. To prevent penetration of the pressurizing fluid (argon) into the graphite, a metal foil bladder was used. The bladders were seamless tantalum tubes 0.002 to 0.003 inches thick. At the test temperatures, tantalum is quite ductile and offers little resistance to deformation. Strains were measured optically using a Physitech Model 440 extensometer aimed at graphite "flags" mechanically attached to the specimen for axial strains, and an Optron Model 800 extensometer aimed at the diametral extremes of the specimen gage section for hoop

strains. The graphite flags were designed to be flexible to accommodate the circumferential growth of the specimen.

Elevated temperatures were obtained in an electric resistance heated furnace (graphite elements) shown in Figure 7. Temperatures were measured optically. The rigid fixture yoke which forms the structural frame of the furnace is cooled with circulating water. The furnace is purged with argon to prevent oxidation.

All tests were conducted at crosshead speeds and pressurization rates which gave an average strain rate (maximum principal strain) between 0.005 and 0.015 per minute. All biaxial tests were under approximately "radial" loading; that is, the pressure was manually controlled to increase in proportion to the axial load so that the ratio of axial to hoop stress was approximately constant up to failure.

Because of the expense of elevated temperature testing the number of specimens tested was necessarily limited. For example only two specimens were tested in some stress states at 3000°F and only one at 4000°F. In an attempt to increase the significance of the data obtained with a limited number of specimens, many of the specimens tested at elevated temperature were first proof tested in the same stress state at room temperature. The proof test was intended to reduce scatter in the high temperature data by eliminating the specimens which might fail significantly below the average strength. The proof test consisted of loading the specimen to a stress level equal to 90% of the average strength obtained with the specimens tested to failure at room temperature. After proof loading the surviving specimens were annealed at 2000°C in inert gas (helium) for one hour to restore the original stress-strain response (Reference 5). After annealing, the specimens were inspected for concentricity and perpendicularity of gage section and critical loading surfaces (Figure 3) to ensure that no distortion occurred as a result of proof loading and annealing; no distortions were observed in this inspection.

RESULTS

Figures 8 through 11 show some typical specimens reassembled after fracture. The fracture initiation sites are usually determinable by

following the crack bifurcation patterns back to the original crack segment. All failures apparently occurred within the gage section of the biaxial specimens. It may be seen that the crack orientation changes with stress-state. When the axial stress is greater than the hoop stress, as in Figure 8, the orientation of the crack is, as expected, circumferential. When the stress ratio is reversed as in Figure 9, the crack runs axially. When the stress ratio is one-to-one, no preferred crack orientation would be expected and, in fact, a variety of orientations were observed; Figure 10 shows an example. In compression-tension stress states the initial crack ran axially (Figure 11) indicating that failure was due primarily to the circumferential tensile stress.*

The fracture stresses measured at room temperature are shown in Figure 12. Several points might be noted:

- 1) There is considerable scatter in the observed strengths. This might be expected from the scatter in uniaxial measurements (Figure 1). This suggests that a failure criterion for graphite should be statistically based on an acceptable risk of failure rather than, as is commonly attempted, on a line drawn through the mean strengths obtained in each stress state.
- 2) The average tensile stress at failure in the biaxial tension quadrant is less than that in uniaxial tension. This implies that the maximum principal stress failure criterion does not really represent the material and that certainly the maximum principal strain theory (which was used in Reference 6, for example) does not apply.
- 3) The compressive stress component seems to have little effect on the maximum tensile stress at failure until it begins to approach the compressive strength of the material.

*Specimens tested at -4:1 ratio of axial compressive stress to hoop stress at room temperature shattered into too many pieces for adequate reconstruction of the crack pattern. This was also true of all specimens tested at elevated temperature.

- 4) Figure 12 includes data obtained with the uniaxial and hoop tension specimens. In tension, the uniaxial specimen gives strength slightly higher than the biaxial specimen. This is tentatively attributed to volume (and/or surface area) effects. In compression the difference between uniaxial and biaxial specimen strengths is attributed to premature buckling of the biaxial specimen wall. The data obtained on biaxial specimens and hoop ring specimens in pure hoop tension are quite similar.
- 5) The hoop stress values presented are based on the thin-wall approximation relating stress to pressure and therefore represent the average hoop stress in the specimen wall. Based on a non-linear stress analysis, the maximum hoop stress (at the inner diameter) is estimated as being less than four percent higher than the thin-wall approximation.

As yet, no analytical development of failure criteria has been attempted using these data. However, as a matter of interest, Figure 13 is presented to show how some available failure criteria fit the room temperature data. It is seen that Weibull's criterion (as presented for hydrostone plaster in Reference 7) fits the median strengths fairly well in the tension-tension quadrant; it predicts the observed decrease in biaxial tensile strength. In highly compressive states the data follow approximately a maximum shear stress (Tresca) criterion. Possibly significant is the suggestion that the lower bound of strengths in the tension-tension quadrant, and in the upper portion of the compression-tension quadrant, are matched fairly well by a maximum principal stress criterion which, in this regime, coincides also with the Griffith criterion (Reference 8). These observations suggest that the behavior in biaxial tension might be explained in terms of the statistics of finding an appropriately oriented flaw within the specimen. Under biaxial tension more flaw orientations are critical to strength than under uniaxial tension;

thus the median biaxial strength is expected to be lower than the median biaxial strength. The lower bound strength, however, is obtained when the most critical flaw is critically oriented (Griffith's theory) so that the effect of stress state is minimized.

Figure 14 shows the strength data obtained at 3000°F, and Figure 15 shows how test temperature affects the biaxial strength data. The strengths at 3000°F and 4000°F, in biaxial stress states, appear to be higher than at room temperature in approximately the same proportion as the increase in uniaxial tensile strength. There appears to be no significant difference between strengths at 3000°F and 4000°F based on the very limited data obtained at 4000°F. In general the remarks made above with respect to the room temperature data apply to the elevated temperature data. However, the elevated temperature data does not extend as far into the compression-tension quadrant because buckling of the specimen tended to occur in the more highly compressive stress states.

Figure 16 shows the strains measured at failure at room temperature. It should be noted that, whereas the stress states are biaxial (except for a small radial compressive component resulting from the internal pressure), the strain states are distinctly triaxial because the radial strain is not negligible. However, in the absence of direct measurement the radial strain must be analytically estimated. Obviously a two dimensional plot, as in Figure 16, cannot present a comprehensive picture of the strain states. Furthermore, the strain data was measured at the exterior surface of the specimen. The hoop strain gradient through the wall thickness is greater than the corresponding stress gradient and may be shown to depend on the biaxial stress state because of Poisson's ratio effects. Therefore the maximum strains experienced by the specimen might be considerably different from the measured values. For example, the hoop strain at the inner diameter is estimated to be between 5 and 30% higher than measured at the outer diameter, depending on the stress-state. With the noted restrictions in mind, it is seen that the trend in strain data is what might be expected, qualitatively, from a consideration of Poisson's effect; that is, the tensile hoop strain at failure

is decreased by the application of axial tension and increased by axial compression. Figure 17 shows the effect of test temperature on the failure strain data. It is seen that the trend at 3000°F is similar to that at room temperature and that strain to failure increases markedly between 3000°F and 4000°F.

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Fig. 1. Cumulative Distribution of Tensile Strength, AXF-5Q.

NOTES

- 16

Fig. 3. Biaxial Test Specimen.

Fig. 4. Biaxial Test Arrangement for Compression-Tension Testing at Room Temperature.

Fig. 5. Schematic of Biaxial Specimen Assembly for Room Temperature Test.

Fig. 6. Schematic of Biaxial Specimen Assembly for Elevated Temperature Test.

Fig. 7. Rigid-Fixture Furnace.

Fig. 8. Reconstructed Specimen: Axial Tensile Stress Greater than Hoop Tensile Stress. Arrow denotes initiating crack. Specimen No. 42-4; axial-to-hoop stress ratio 2:1; tested at room temperature.

Fig. 9. Reconstructed Specimen: Axial Tensile Stress Less than Hoop Tensile Stress. Specimen No. 43-5; axial-to-hoop stress ratio 1:2; tested at room temperature.

Fig. 10. Reconstructed Specimen: Axial Tensile Stress Equal to Hoop Tensile Stress. Specimen No. 69-4; axial-to-hoop stress ratio 1:1; tested at room temperature.

Fig. 11. Reconstructed Specimen: Axial Compressive Stress Equal to Hoop Tensile Stress. Specimen No. 26-3; axial-to-hoop stress ratio -1:1; tested at room temperature.

Fig. 12. Biaxial Fracture Stresses at Room Temperature, AXF-5Q Graphite.

Fig. 13. Some Failure Criteria Applied to AXF-5Q Biaxial Data.

Fig. 14. Biaxial Fracture Stresses at 3000°F, AXF-5Q Graphite.

Fig. 15. Effect of Test Temperature on Biaxial Fracture Stresses of AXF-5Q Graphite.

Fig. 16. Biaxial Strains Measured at Fracture of AXF-5Q Graphite at Room Temperature.

Fig. 17. Effect of Test Temperature on Biaxial Fracture Strains, AXF-5Q Graphite.

✓ GEOMETRICAL PARAMETERS AFFECTING THE
FRACTURE TOUGHNESS OF GRAPHITE*

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ABSTRACT

The fracture mechanics approach for predicting brittle fracture has come to be widely accepted as a valuable technique for structural design. Since graphite fails in a brittle manner, the application of fracture mechanics to this material is natural. The results of a study of the effects of several geometrical parameters on the stress-intensity factor at onset of rapid crack propagation, K_{IC} , are reported. For plane-strain fracture, K_{IC} is more specifically referred to as K_{IC} , the material property used in fracture mechanics analyses. Suitable specimen designs for measurement of K_{IC} for graphite were determined. Several specimen configurations were tested from two graphites, ATJ and AXM.

The effects of specimen size, specimen width, notch sharpness, and notch depth on the values of K_{IC} obtained from tests of notched beams in four-point bending were investigated. The most critical geometrical parameter of the notched beams was found to be notch sharpness. Beams as small as 4 in. long by 3/16 in. wide by 7/16 in. deep were found to give valid K_{IC} values when the notch was sufficiently sharp.

Values of K_{IC} were also determined from three other types of specimens: notched tensile specimens, double cantilever beams, and compact tension specimens. The values of K_{IC} from the different specimen types are compared, and recommendations are made regarding suitable specimen geometries for measuring the fracture toughness of graphites when the specimen size is limited.

The use of fracture mechanics to predict the strength of specimens which fracture at naturally occurring flaws rather than at gross machined notches was also explored. The flaws that would produce fracture at the loads that broke unnotched specimens of ATJ and AXM graphites are comparable in size to flaws observed in photomicrographs of these materials.

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Fracture mechanics has come into widespread use for predicting brittle fracture, especially for applications involving high-strength metals. A basic postulate of fracture mechanics is the existence of flaws in the material which raise the local stress level and produce fracture when the nominal stress level reaches a certain value. Graphite is a relatively brittle material; only a small amount of plastic deformation occurs before graphite fractures. Graphite contains many tiny flaws. Therefore, application of the fracture mechanics approach to predicting fracture of graphite should be quite suitable.

The measure of fracture toughness used in this study is the stress-intensity factor at onset of rapid crack propagation, K_{Ic} , which has the units $\text{ksi}/\sqrt{\text{in.}}$. Elastic stress analyses are used to establish the relationship between applied load, crack length, and stress-intensity factor for each specimen geometry. The value of K_{Ic} is then determined from the crack length and load at fracture.

Fracture toughness is a minimum under plane-strain conditions. Furthermore, under plane-strain conditions the mode of fracture is a wedge-opening of the crack, which is customarily referred to as mode I. The stress-intensity factor at onset of rapid crack propagation under plane-strain conditions is referred to as K_{Ic} . The controlling parameter for design to avoid brittle fracture is generally K_{Ic} .

The purpose of this study was to examine the effects of variations in geometry on the measured value of K_{Ic} so that valid values of K_{Ic} for graphites could be obtained for use in design. Two graphites, ATJ and AXM, were used in these studies. ATJ graphite is a commercial premium grade, molded graphite which has a maximum grain size of 0.006 in. It has been produced by Union Carbide Corporation, Carbon Products Division, for a number of years, and its mechanical behavior has been well characterized. It has a density of 1.73 g/cc and room temperature tensile strengths of 3800 and 3000 psi in the with-grain and across-grain directions, respectively. AXM graphite is an extremely fine grain, isotropic, premium quality graphite manufactured by POCO Graphite, Inc. It has about the same density as ATJ graphite, and it has a tensile strength of 7400 psi.

Such geometrical factors as specimen size, specimen width, notch sharpness, and notch depth were examined. Several types of specimens were tested, as shown in Fig. 1. The notched beam tested under four-point bending was the basic specimen used in these studies. There were two sizes of notched beams: the large beams were 12 in. long, $1\frac{1}{4}$ in. deep, and $\frac{1}{4}$, 1, and $1\frac{1}{2}$ in. wide; the small beams were 4 in. long, $\frac{7}{16}$ in. deep, and $\frac{3}{16}$ in. wide. Several notch shapes and notch depths were used in the beams. The notched tensile specimens were 12 in. long and were $\frac{5}{8}$ in. in diameter in the gage section; V-notches with a 45° included angle were machined to three notch depths.

The details of the double-cantilever beam specimen are shown more clearly in Fig. 2. Special clevises were attached to the specimen with pins through the holes in the specimen. An initial notch, about $\frac{1}{2}$ in. long, was machined in the specimen. Narrow flat-bottomed grooves on both sides were used to force the crack to propagate along the length of the specimen. The compact tension specimen is shown more clearly in Fig. 3. This type, approximately $\frac{7}{16} \times \frac{7}{16} \times \frac{3}{16}$ in., is the smallest of the specimens.

The different types of notch tips used in the notched beam tests are shown in Figs. 4-8, all of which are the same magnification. The notch width is considered to be the measure of notch sharpness. The dulllest notch, shown in Fig. 4, is 0.033 in. wide and has a flat bottom. This notch was made with a slitting saw. The crack always started at one of the corners, as would be expected. Figure 5 shows the V-notch with a 45° included angle; the root radius of these notches varied from 0.0006 to 0.010 in. Figure 6 shows the narrowest slot that we could make with a slitting saw; this slot is 0.005 in. wide. Figure 7 shows the sharpest notch that we can machine. A 0.020-in. slot is machined; a cutter is then used to make a 30° V-notch at the bottom of the slot. The root radius of the notch is approximately 0.0005 in. A true crack is shown in Fig. 8. This crack was produced in one of the large notched beams by driving a chisel into the beam with a hammer. A really satisfactory method for producing such a crack and for locating its tip has

not been found. The tip of the chisel crack could be identified in the AXM beams after the specimen had been broken, as shown in Fig. 9. The large AXM beams with chisel cracks were the only set of specimens with cracks in which the crack tip could be accurately located.

Let us now examine the effects of the various notch tip shapes on fracture toughness, plotted in Fig. 10. The square root of the notch width at the crack tip was used as a quantitative measure of notch sharpness and is the abscissa of the graph. The ordinate is the K_{Ic} value. Average results from all the notched beam tests are shown for both AXM and ATJ graphite. The 1-1/2-in.-wide large notched beams had three types of notches: cracks, 0.005-in.-wide slots, and 0.033-in.-wide slots. For the AXM beams, where the tips of the cracks could be accurately located, the K_{Ic} value decreased continuously as the crack width decreased. For the ATJ beams the wide slot gave a higher average K_{Ic} value, but so did the crack.

Looking now at the results from the small beams, we see that again fracture toughness decreased as the notch became sharper, except for the cracks. The small AXM beams with 0.006-in. slots gave high K_{Ic} values for some undetermined reason. It is evident from this mass of data that notch sharpness has an influence on the measured value of fracture toughness of graphite and that one should use the sharpest possible notch providing the tip can be accurately located. Inability to accurately locate the crack tip makes the use of true cracks as initiating flaws undesirable. The difference in the K_{Ic} values obtained with 0.001-in. slots and 0.005-in. or 0.006-in. slots is small. It is concluded that machined slots which are 6 mils or less in width at the tip are adequate for measuring the fracture toughness of graphite.

Let us consider the effect of the depth of the notch on fracture toughness. Figure 11 shows K_{Ic} values from large notched beams of ATJ graphite with five depths of notches, from 1/16 to 5/8 in. deep. The value of K_{Ic} is plotted as a function of the ratio of notch depth to total beam depth. Notches more than 1/8 the beam depth gave consistent values of K_{Ic} for ATJ graphite; shallower notches gave low K_{Ic} values. Figure 12

shows the effect of notch depth on the K_c value for AXM graphite. The notch depth did not affect the K_c value for AXM graphite except for a c/d ratio of 0.05. It is therefore concluded that the fracture toughness of AXM graphite can be measured with beams that have c/d ratios from $1/8$ to $1/2$ and that the fracture toughness of ATJ graphite can be measured with beams that have c/d ratios from $1/4$ to $1/2$.

Now we will consider the effect of specimen width on the fracture toughness measured on large notched beams. Figure 13 shows average K_c values for different notches in AXM and ATJ graphite beams plotted as a function of the beam thickness or width. AXM beams with 0.005-in. slots indicate a decrease in fracture toughness as the beam width is increased. AXM beams with 0.033-in. slots and ATJ beams with 0.005-in. and 0.033-in. slots indicate an increase in K_c with an increase in beam width. It is concluded that there is no effect of beam thickness on the value of K_c in the range from $1/4$ in. to $1-1/2$ in.

These results lead to the conclusion that valid K_{Ic} values for graphite can be obtained using beams that have slots that are no more than 0.006 in. wide and are sufficiently deep. The measured values of K_{Ic} for AXM and ATJ graphites are given in Table 1. The value of K_{Ic} for AXM graphite (which is isotropic) is $1.17 \text{ ksi} \sqrt{\text{in.}}$ with a standard deviation of $0.08 \text{ ksi} \sqrt{\text{in.}}$. ATJ graphite is transversely isotropic and therefore has three principal systems of crack propagation. When the stress is applied in the with-grain direction, the K_{Ic} value is the same whether the crack propagates in the with-grain direction or the across-grain direction. For ATJ graphite loaded in the across-grain direction, the fracture toughness, K_{Ic} , is $0.69 \text{ ksi} \sqrt{\text{in.}}$ with a standard deviation of $0.04 \text{ ksi} \sqrt{\text{in.}}$.

Next we will compare the results from the different types of specimen. Figure 14 shows the average K_c value for each type of specimen. The K_c values for the beams are the K_{Ic} values from specimens with slots that were sufficiently sharp and deep, as discussed previously. The values of K_c from different specimens are in good agreement with the K_{Ic} values from large notched beams, except for the double-cantilever beam specimens and the ATJ notched tensile specimen. It is concluded that the large notched beam, small notched beam, and compact tension specimen are good specimens for measuring the fracture toughness of graphite.

It has been clearly indicated that fracture mechanics applies to graphite specimens with gross machined notches. However, one would not purposefully machine such notches in a structural component; the notches severely weaken the graphite. Two 12-in.-long by 1-1/2-in.-wide by 1-1/4-in.-deep unnotched AXM beams broke at loads of 2120 psi and 2190 psi. Similar beams with 0.005-in.-wide slots only 1/6 in. deep broke at loads of only 611 psi and 631 psi. No one would purposefully reduce the strength of a structure by that amount. So the question is, "Can fracture mechanics be used in the design of components in which the flaws are the inherent innumerable small random defects and imperfections rather than a single gross flaw?"

The information that we will examine now indicates that indeed it probably can. The strength of unnotched beams of AXM and ATJ graphites and the values of K_{Ic} from the notched beams were substituted into the same equation that had been used to determine K_{Ic} for the notched beams knowing their fracture load and notch depth, and the equation was solved for notch depth. The notch depth calculated for ATJ graphite was approximately 0.010 in. The notch depth calculated for unnotched AXM beams was approximately 0.003 in. Are the natural flaws in these two materials this size? Figure 15 indicates that they are. A photomicrograph of ATJ graphite is shown on the left. The photomicrograph on the right is to the same magnification and is of AXM graphite. The calculated flaw sizes are indicated by the white marks on the micrographs. As can be observed, the largest natural flaws in both materials are about the size of the calculated flaws. This indicates that fracture mechanics can probably be applied to graphite components which do not contain gross machined flaws.

Table 1. Plane-Strain Fracture Toughness Values
for AXM and ATJ Graphites

Graphite	Loading Direction	Cracking Direction	K_{Ic} (ksi $\sqrt{\text{in.}}$)	
			Mean	Standard Deviation
AXM			1.17	0.08
ATJ	With-grain	With-grain	0.82	0.05
ATJ	With-grain	Across-grain	0.81	0.06
ATJ	Across-grain	With-grain	0.69	0.04

Fig. 1. Fracture Mechanics Specimens.

Fig. 2. Double Cantilever Beam Specimen.

Fig. 3. Compact Tensile Specimen.

Fig. 4. Slot, 0.033 in. Wide.

Fig. 5. V-Notch.

Fig. 6. Slot, 0.005 in. Wide.

Fig. 7. Machined Notch with 0.0005-in. Root Radius.

Fig. 8. Crack Produced by Chisel in AXM Graphite Beam.

Fig. 9. Fracture Surface of AXM Graphite Beam with Chisel Crack.

Fig. 10. Effect of Notch Sharpness on Fracture Toughness.

Fig. 11. Effect of Notch Depth on Fracture Toughness of ATJ Graphite.

Fig. 12. Effect of Notch Depth on Fracture Toughness of AXM Graphite.

Fig. 13. Effect of Specimen Thickness on Fracture Toughness.

Fig. 14. Comparison of K_c Values from Several Fracture Toughness Specimens.

ATJ

AXM

Fig. 15. Photomicrographs of ATJ and AXM Graphites, Showing Comparison of Calculated Sizes of Natural Flaws with Actual Flaws.

X APPLICATION OF WEIBULL STATISTICAL THEORY
TO THE STRENGTH OF REACTOR GRAPHITE

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ABSTRACT

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The Weibull statistical theory for the strength of brittle materials has the potential for predicting failure probabilities in structures of any volume or stress state, including multi-axial stresses and stresses which vary with position. The Weibull equations were used to calculate failure probabilities in four-point bend-test samples and in internally pressurized thick-walled tubes with either zero axial stress or with a super-imposed axial tensile stress. The calculations predict that the ratio of the median strength of samples with non-uniform stress systems, σ_{nu} , to the median tensile strength, σ_t , is given by expressions of the form:

$$\frac{\sigma_{nu}}{\sigma_t} = \left(\frac{V_t}{V_{nu}} \right)^{\frac{1}{m}} \times (\text{stress distribution term})$$

where V_t and V_{nu} are the volume of the tensile samples and non-uniform stress samples respectively, and m is the Weibull modulus. The stress distribution term depends on the Weibull modulus, the way the stress varies with position, and the biaxial stress ratio within a small volume element of material.

Some predictions of the Weibull theory were tested against the results of tensile tests, bend tests, and tube-burst tests with and without superimposed tensile stress, performed on H-327 graphite. It was found that the distribution of strength values among a group of tensile samples with the same orientation and log location is described equally well by a Weibull or a Gaussian distribution. The strength measured from the tube-burst tests showed the expected dependence on wall thickness, but the absolute strength values were higher than predicted by Weibull theory. The moduli of rupture obtained from the bend tests were lower than the theoretical predictions. However, when the volume-ratio term in the above equation was set equal to 1 and the stress-distribution term alone was used, the calculated values were in reasonable agreement with the observations for all series of tests. Thus, the Weibull equations correctly predicted the changes in failure probability due to non-uniform stress distributions, but over-estimated the dependence on sample volume.

INTRODUCTION

Strength measurements on brittle materials show a high variability and a dependence on the physical size and type of the test sample. This can result in serious errors when conventional design methods are used and strength values from a limited number of tensile tests are used in conjunction with stress analysis to predict failure in large reactor core components subject to complex stress states. For example, the method of selecting a "safe working stress" that is some fraction of the yield stress, which is adequate for metal structures, cannot be applied to brittle materials with a statistical spread in strength values, because there will be a finite probability of failure for any applied stress above zero. Similarly, it may not be sufficient to predict a failure probability based on the mean fracture stress and standard deviation for a few tensile tests, because the implicit assumptions that the strength values are distributed normally and the fracture stress is independent of sample volume and stress state may not be justified.

One design approach for brittle materials is to use the Weibull statistical theory of strength, in which the strength of a body under a tensile stress is assumed to be controlled by the distribution of surface or internal flaws. This theory has the potential for predicting failure probabilities in structures of any volume and stress state, including multi-axial stresses and stresses which are a function of position. The Weibull theory has been found to be applicable to many ceramic materials, including some graphites. [1-6].

Recent test data obtained on Great Lakes Carbon Company H-327 graphite [7-9] showed significant differences between strength values derived from tensile tests, bend tests, tube-burst tests with a range of wall thicknesses, and biaxial tests in which samples representing the material surrounding a fuel hole in a High-Temperature Gas-Cooled Reactor graphite block were subjected to a tensile load and a simultaneous internal pressure. This report describes work which applies the Weibull equation to these stress states, and compares the test results with the theoretical predictions.

THEORY

Form of the Weibull Distribution

Weibull's theory of the strength of brittle solids [10,11] assumes that the material contains a distribution of flaws, either on the surface or throughout the volume. When the material is subjected to a tensile stress, the combination of the highest local stress concentration with the most serious flaw controls the strength of the bulk (the "weakest link hypothesis"). Weibull arbitrarily took the survival probability, $S(\sigma)$, of a small volume element of material under a stress σ to have the form:

$$\ln[S(\sigma)] = - \left(\frac{\sigma - \sigma_u}{\sigma_o} \right)^m$$

This yields the following expression for the survival probability of the whole sample, S_V :

$$S_V = \exp \left[- \int_V \left(\frac{\sigma - \sigma_u}{\sigma_o} \right)^m dV \right] \quad (1)$$

where σ is the tensile stress in the element dV , σ_u is the "minimum failure stress" below which the failure probability is zero, σ_o is a normalizing parameter (for a uniformly loaded sample of unit volume, an applied stress of $\sigma_o + \sigma_u$ would result in a survival probability of $1/e = 0.368$), and m is a parameter known as the Weibull modulus. In practice, σ_u , σ_o and m for a particular material are found from experimental strength measurements by best-fit methods. The accuracy with which σ_u can be obtained is low; analysis of some data batches gives a negative best-fit value for σ_u , which is physically meaningless. For materials such as graphite, σ_u may be taken as equal to zero; analysis of strength data on Poco graphite [5] indicates that there is no significant difference in the fit of the experimental data between taking the best-fit value for σ_u and taking $\sigma_u = 0$. In practical terms, this means that there is no stress below which the probability of failure is zero. For the calculations described in this report a value of zero was assumed for σ_u .

When $\sigma_u = 0$ and the stress is uniform through the sample volume, Eq. (1) may be written as:

$$\ln \ln \left(\frac{1}{S_V} \right) = \ln V - m \ln \sigma_o + m \ln \sigma \quad (2)$$

Thus, the value of m may be obtained from a batch of strength data from the slope of a plot of $\ln \ln(1/S)$ versus $\ln \sigma$. Other methods for estimating m from experimental data are given in Ref. 12.

The strength distribution given in Eq. (1) predicts higher probabilities of failure at stresses well removed from the mean strength than predicted by a normal distribution of strengths. Fig. 1 shows a comparison between the probability density functions for Weibull distributions with $m = 5$ and $m = 10$, and normal distributions with the same standard deviations.

Effect of Sample Volume

For tensile samples where $\sigma_u = 0$ and the stress is uniform throughout the sample volume, Eq. (1) gives the survival probability as:

$$S_V = \exp \left[- V \left(\frac{\sigma}{\sigma_o} \right)^m \right] \quad (3)$$

where V is the load-bearing volume. For two batches of samples of volume V_1 and V_2 , respectively, the condition for equal failure probability is:

$$V_1 \left(\frac{\sigma_1}{\sigma_0} \right)^m = V_2 \left(\frac{\sigma_2}{\sigma_0} \right)^m$$

$$\therefore \frac{\sigma_1}{\sigma_2} = \left(\frac{V_2}{V_1} \right)^{\frac{1}{m}} \quad (4)$$

Thus the median strength of the low-volume samples is higher than that of the high-volume samples, and the sensitivity to sample volume is greatest for low values of m .

Non-uniform Stress Distribution: Bending

For an elastic material, the stress in the center span of a sample under four-point loading varies with the distance x from the mid-plane, and for the part of the beam between the inner and outer knife edges, the stress also varies with the distance from the outer knife edge. Applying Eq. (1) to this stress distribution gives:

$$S_v = \exp - \left(\frac{\sigma_{\max}}{\sigma_0} \right)^m \left(\frac{V_{cs}}{2(m+1)} + \frac{V_{os}}{(m+1)^2} \right)$$

where V_{cs} is the volume of the center span of the sample (between the two inner knife edges) and V_{os} is the volume of the outer span (between the inner and outer knife edges). Thus the distribution function for the modulus of rupture of a batch of bend-test samples is similar to that for tensile strengths [Eq. (3)], differing only in the volume term. For a tensile sample of the same material under a tensile stress σ_t , and with a volume V_t , the survival probability [Eq. (3)] is:

$$S_{V(\text{tens.})} = \exp \left[- V_t \left(\frac{\sigma_t}{\sigma_0} \right)^m \right]$$

Therefore the condition for equal survival probability for bend-test samples and tensile samples is given by:

$$\frac{\sigma_{\max}}{\sigma_t} = [2(m+1)]^{\frac{1}{m}} \left[\frac{V_t}{V_{cs} + \frac{2}{(m+1)} V_{os}} \right]^{\frac{1}{m}} \quad (5)$$

For a circular cross-section beam of radius r , subjected to four-point loading, the corresponding equation (for integer values of m) is:

m even:

$$\frac{\sigma_{\max}}{\sigma_t} = \left[\frac{2 \times 4 \times 6 \cdots (m+2)}{1 \times 3 \times 5 \cdots (m-1)} \right]^{\frac{1}{m}} \left[\frac{V_t}{V_{cs} + \frac{2}{(m+1)} V_{os}} \right]^{\frac{1}{m}} \quad (6a)$$

m odd:

$$\frac{\sigma_{\max}}{\sigma_t} = \left[\frac{1 \times 3 \times 5 \dots (m+2)}{2 \times 4 \times 6 \dots (m+1)} \times \frac{\pi}{2} \right]^{\frac{1}{m}} \left[\frac{V_t}{V_{cs} + \frac{2}{(m+1)} V_{os}} \right]^{\frac{1}{m}} \quad (6b)$$

Eqs. (5) and (6) predict the relationship between the median modulus of rupture of a group of bend samples and the median ultimate tensile strength of a group of tensile samples of the same material. If the relative volumes of the samples are such that $V_t = V_{cs} + [2/(m+1)] V_{os}$, the volume ratio term is equal to 1 and the strength ratio is given by the first term in square brackets in Eqs. (5) and (6). The value of this strength ratio is plotted as a function of m in Fig. 2.

Internally Pressurized Thick-Walled Cylinder (Zero Axial Stress)

The Weibull treatment for an internally pressurized thick-walled cylinder may be derived from the general Weibull equations and the Lamé equation relating the tangential stress in the tube wall to the internal pressure. The first case applies to a cylinder free from axial stresses. Elastic behavior is assumed.

The condition for equal survival probability causes the ratio of the median inner-surface tangential stress at fracture, $\sigma_{\max}$, to the median tensile strength, σ_t , of tensile samples of volume V_t , to be:

$$\frac{\sigma_{\max}}{\sigma_t} = \left(\frac{V_t}{V_{cyl}} \right)^{\frac{1}{m}} \left[\frac{(x^2 - 1)(x^2 + 1)^m}{x^2 \cdot f(x, m)} \right]^{\frac{1}{m}} \quad (7)$$

where x = ratio of the outside diameter to the inside diameter and V_{cyl} is the volume of the material in the cylinder wall. $f(x)$ is given by:

$$f(x) = \left[1 - \frac{1}{x^2} + 2m \ln x + \binom{m}{2} (x^2 - 1) + \dots - \frac{1}{(n-1)} \binom{m}{n} (x^{2n-2} - 1) \dots + \frac{1}{(m-1)} (x^{2m-2} - 1) \right]$$

Thus the ratio of the median tube-burst strength to the median tensile strength is given by the product of a "volume ratio" term and a "strength ratio" term. The "strength ratio" term,

$$\left[\frac{(x^2 - 1)(x^2 + 1)^m}{x^2 \cdot f(x, m)} \right]^{\frac{1}{m}}$$

was evaluated for values of x = (O.D./I.D.) from 1.0 to 4.0, and values of m from 2 to 20. The results are shown in Fig. 3.

Biaxial Stress System

The general relationships required to extend the treatment to biaxial tensile stresses are given in Weibull's original paper [10]. If the normal tensile stress is the only stress element contributing to the risk of failure within a volume element, the variation of the normal stress with angle should be taken into account and the expression for the probability of survival is obtained by integrating over half a sphere:

$$S_V = \exp \left[- \int_V \int_0^{\frac{\pi}{2}} \int_0^{2\pi} \left(\frac{\sigma_n}{\sigma_0} \right)^m \cos \phi \, d\psi \, d\phi \, dV \right] \quad (8)$$

where ϕ and ψ are the angles of latitude and longitude in the usual coordinate system, and σ_n the normal component of stress, acting in the direction (ϕ, ψ) . Eq. (8) holds when the stress is tensile in all directions; if the stress is compressive in any direction the limits of integration are modified to encompass only the tensile region.

Equating the failure probability for equal uniformly stressed volumes under a uniaxial stress, σ_z , and a biaxial stress with maximum principal stress σ_x , and stress ratio, $\sigma_y/\sigma_x = R$, gives the ratio of median failure stresses as:

$$\frac{\sigma_x}{\sigma_z} = \left[\frac{2\pi}{(2m+1) \int_0^{\frac{\pi}{2}} \int_0^{2\pi} \cos^{2m+1} \phi (\cos^2 \psi + R \sin^2 \psi)^m \, d\psi \, d\phi} \right]^{\frac{1}{m}} \quad (9)$$

A computer program was written to evaluate the strength ratio, σ_x/σ_z , for biaxiality ratios between 0 and 1 and m values between 2 and 20. The results are shown in Fig. 4 in the form of failure envelopes for various values of σ_x and σ_y . The results predict that failure under biaxial stress would occur at stress levels considerably below the stress levels for the maximum principal stress failure criterion normally assumed for brittle materials. The envelopes approach the maximum principal stress envelope as $m \rightarrow \infty$.

Internally Pressurized Cylinder With Axial Stress

In a conventional tube-burst test in which the gas pressure acts on the closed end of the tube, and also in tests in which a tensile load is superimposed on an internally pressurized tube sample, the material in the tube wall is subject to biaxial tensile stress. The failure conditions are therefore modified from those calculated in the section entitled Internally Pressurized Thick-Walled Cylinder for zero axial stress. The expression for survival probability is obtained by combining Eq. (8) with the Lamé equation for tangential stress. For equal failure probability of failure in the tube and in a tensile sample, the ratio of tube-burst strength, $\sigma_{\max}$, to tensile strength, σ_t , is given by:

$$\frac{\sigma_{\max}}{\sigma_t} = \left(\frac{v_t}{v_{\text{cyl}}} \right)^{\frac{1}{m}} (x^2 + 1) \left(\frac{\pi a^2 (x^2 - 1)}{(2m + 1)} \right)^{\frac{1}{m}} \quad (10)$$

$$\left\{ \int_a^b r \left(1 - \frac{b^2}{r^2} \right)^m \left[\int_0^{\frac{\pi}{2}} \int_0^{2\pi} \cos^{2m+1} \phi (\cos^2 \psi + R \sin^2 \psi)^m d\psi d\phi \right] dr \right\}^{-\frac{1}{m}}$$

where a is the internal radius and b is the external radius. The value of the double integral (the biaxiality term) may be calculated for the biaxiality ratio, R , appropriate to the radial position in the cylinder.

A computer program was used to evaluate the ratio $\sigma_{\max}/\sigma_t$ from Eq. (10) for a closed-end tube with zero superimposed stress. The results for equal-volume tensile samples and tube-burst samples are shown in Fig. 5 for values of m between 2 and 16, and values of x (O.D./I.D.) between 1 and 4. The strengths of the cylinders are reduced below the values for zero axial stress (Fig. 3) with the reductions being greatest for low values of m and of x . For thin-walled cylinders (x approaching 1) the stress in the tube wall becomes uniform and the strength reduction is equal to that predicted for a biaxial stress ratio of 0.5 (Fig. 4).

TEST PROCEDURES

Tests were carried out at room temperature on H-327 graphite representing pre-production and production fuel block material [7-9] for the Fort Saint Vrain High-Temperature Gas-Cooled Reactor. Tensile tests were performed on 3" long by 0.5" diameter cylinders. Four-point bend tests were made both on rectangular cross-section samples and circular cross-section samples. For the rectangular samples the knife edges were equally spaced 0.875" apart, the sample thickness was 0.17", and the sample width was either 0.25", 0.5", or 1.0". For the cylindrical samples the knife edges were equally spaced 0.45" apart and the samples were 0.105" diameter. A series of thick-walled tubes were tested by internal pressurization with zero axial stress. The length of the samples was 4.5" and the internal diameter was 0.502"; the wall thickness ranged from 0.086" to 0.500". A number of samples simulating a section surrounding a fuel hole in a PSC fuel block were tested by pre-loading in tension under loads of 0 to 1150 lb and internally pressurizing the fuel hole until the sample failed at a web. The shape of the latter samples is shown in Fig. 6.

Mechanical testing was carried out on an Instron TTC-M-1 machine using a cross-head speed of 0.005 in./min for the uniaxial tensile tests and 0.001 in./min for the beam bend tests. The tensile test specimens with the PSC fuel block hole configuration and also the standard cylindrically shaped pieces were cemented with epoxy resin to a flexible steel coupling mounted in the test machine as per ASTM Test Procedure No. ASTM C 565.

"Hoop stress only" tests were applied by hydraulically pressuring a close fitting rubber hose in the bore of the assembly which remained free to expand within the test length along the axial direction. For biaxial testing a combination of the above tensile and hydraulic tests was applied.

APPLICATION OF THEORY TO EXPERIMENTAL RESULTS

Distribution of Tensile Strengths

A total of 157 tensile strength measurements were available on 3" long by 0.5" diameter cylinders of H-327 graphite [7-9]. These measurements were on samples from nine different logs and included parallel- and perpendicular-cut samples from edge, axial, and mid-radius positions. Samples from both ends and from the mid-length regions of the log were included. Sample locations are illustrated in Fig. 7. The data are shown in Table 1. Within the limitations imposed by the small number of duplicate tests, application of standard statistical tests showed no evidence for any significant difference in strength between samples from similar positions in different logs, between edge samples from the ends and mid-length positions, or between samples from axial and mid-radius positions (perpendicular samples only). Parallel samples were significantly stronger than perpendicular samples from equivalent log positions, and for both orientations samples from the axial mid-length positions (location 6, Fig. 7) were significantly weaker than samples from the edges (locations 1 and 4) or axial-end positions (location 3) (confidence levels > 99.5%). Parallel samples from axial-end positions (location 3) tended to be weaker than those from edge positions (locations 1 and 4) ($t = 2.8$, confidence level = 99%), but for perpendicular samples the difference between these two positions was not significant ($t = 1.3$). It would require a larger number of duplicate tests to establish the significance (if any) of other independent variables.

The variations in strength with position in the log are due principally to differences in the effectiveness of the impregnation process. Pores in the material near the edges and, to a lesser extent, the ends of the logs become filled with impregnant carbon, whereas those in the central regions do not. This results in consistently lower strengths for both parallel and perpendicular samples from the central regions of the logs.

The strength data were lumped into the following categories: parallel, edge of block (locations 1 and 4, Fig. 7); parallel, center, end of block (location 3); parallel, center, mid-length (location 6); perpendicular, edge of block (locations 1 and 4); perpendicular, center and mid-radius, end of block (locations 2 and 3); and perpendicular, center and mid-radius, mid-length (locations 5 and 6). A computer program was used to rank the strength data in increasing order and compute the mean and median values and standard deviation. When N strength values are arranged in ascending order, the best estimate of the failure probability corresponding to the j^{th} strength values is given by [12]:

$$F = \frac{j - 0.3}{N + 0.4} \quad (11)$$

and the corresponding survival probability, S , is $1-F$. Thus, data arranged in this way are suitable for graphical testing against any theoretical distribution which predicts the relationship between stress σ and survival probability, S . In the case of the Weibull distribution, Eq. (2) predicts that a plot of $\ln \ln 1/S$ against $\ln \sigma$ will be a straight line of slope m . A subroutine was included in the present program to use this relationship to obtain the least-squares value of m .

Table 1. Tensile Strength Measurements on H-327 Graphite (psi)

Log Number	Parallel Orientation				Perpendicular Orientation					
	Edge, End Location 1	Edge, Mid-length Location 4	Center, End Location 3	Center, Mid-length Location 6	Edge, End Location 1	Edge, Mid-length Location 4	Mid-radius, End Location 2	Mid-radius, Mid-length Location 5	Center, End Location 3	Center, Mid-length Location 6
114 (Pre-production)	2270 2430 2120 1670	2220 2010	2060 2200 1870 1710	1300 1340	1170 1260 1010 1180	1100 1200	-	-	1110 1010 1000 1090	860 1140
694 (Pre-production)	-	-	-	-	1510 1450 1420 1360 1380 1400	1410 1550 1410 1340 1300 1210	1320 1040 1130	1240 1000 1060	1210 1270	1160 990
2479 (Pre-production)	2370 2430	2630 2330	1820 1770	1460 1460 1200	1350 1310	1300 1450	- -	- -	1250 1510	950 865
83-9976 (Production)	1995	-	1716	-	1257		-	-	1067	
42-2163 (Production)	2280	-	1995	-	1210		-	-	1257	
42-1960 (Production)	2670 2640	2520 2240	1320 1270	1200 1140	1300 1250	1020 1000	-	-	1400 1380	830 723
42-2034 (Production)	2566 2214	2667 2566	2290 2097	1920 1810	1678 1600	1359 1344	-	-	1760 1580	986 960
42-1972 (Production)	2290 1160 2340 2250 2540* 2525*	2160 2130	2340 2250 2540 2525 2150* 2130*	1480 1340	1460 1420	1150 1060	-	-	1100 1080	950 890
42-1790 (Production)	2520 2170 2400	2550 2290	2220 2080 2160	1760 1060 1380 1650 1590 1610	1370 1340 1340	1370 1370 1370	-	-	1600 1300 1400	1049 910 1016 1010 686 986

* Samples taken 1/4 of length along log.

A summary of the strength data is shown in Table 2, together with the corresponding least-square estimates of m and the computed standard deviations. The m -values varied between 6.3 and 11.6, with a weighted mean of 8.5. The highest m -values came from parallel and perpendicular samples from the edges of the logs, suggesting that effective impregnation reduces the spread in strengths in addition to increasing the mean strength. Figs. 8 and 9 are examples of the survival probability plotted on probability paper against stress (normalized to the median strength) for the strongest and the weakest groups of samples. The continuous lines on the plots are the theoretical strength distributions for (a) a normal distribution, and (b) a Weibull distribution, using the computed best-fit parameters. It may be seen that the scatter in the data is too great to decide which of the two distributions fits the data best. For the observed values of m , the two curves only deviate to an appreciable extent at low failure probabilities ($\sim 5\%$). Thus it would require a much larger number of duplicate tests (> 100) to distinguish between the two forms of distribution. Even then it would be very important not to reject any low-strength measurements as "bad samples", because discrimination between the two distributions would rest heavily on the few lowest strength measurements.

Bend Tests

A few four-point tests were performed on parallel and perpendicular rectangular samples of H-327 graphite.

The moduli of rupture are plotted against the ultimate tensile strength in Fig. 10. The moduli of rupture are consistently higher than the tensile strengths, but there is no systematic dependence on sample width. The ratio between modulus of rupture and tensile strength predicted by the Weibull theory is given in Eq. (5), where the first term is the volume-ratio term and the second term is the stress-distribution term. The predicted moduli of rupture were calculated from Eq. (5), assuming a Weibull modulus of 8, and are shown in Fig. 10. The predicted values using Eq. (5) tend to fall higher than the observed values, especially for the low-width samples. The values calculated from the stress distribution term alone fall closer to the mean of the experimental values, although the scatter in the data and the small number of tests make it difficult to establish a good correlation.

A second series of four-point bend tests was performed on 0.105" diameter cylindrical samples. Fig. 11 shows the moduli of rupture plotted against the mean ultimate tensile strength values of companion samples.

The appropriate Weibull equation predicting the relationship between the modulus of rupture of cylindrical samples and the UTS is Eq. (6). The relationship of Eq. (6) is shown for $m = 8$ in Fig. 11, together with that of the stress-distribution term alone (i.e., the relationship predicted for a volume-ratio term of 1). As in the case of the rectangular samples, the data points fall lower than the relationship predicted by the full equation, but are in good agreement with that obtained using the stress-distribution term alone.

Thick-Walled Tube-Burst Samples

A series of thick-walled tubes of H-327 graphite with their axes parallel to the extrusion direction were tested by internal pressurization with zero axial stress. The length of the samples was 4.5" and the internal diameter was 0.502"; the wall thickness ranged from 0.086" to 0.500". The inner surface tangential stresses at fracture are plotted as a function of outside diameter to inside diameter ratio in Fig. 12. The fracture stresses increase with increasing O.D./I.D. ratio and are consistently higher than the tensile strength of perpendicular-cut samples from the same location in the same log.

Table 2. Summary of Tensile Strength Measurements on H-327 Graphite

Property	Parallel Orientation			Perpendicular Orientation		
	Edge of Block Locs. 1 and 4 ^a	Center, End-of-Block Loc. 3	Center, Mid-Length Loc. 6	Edge-of-Block Locs. 1 and 4	Center and Mid- Radius, End-of-Block Locs. 2 and 3	Center and Mid- Radius, Mid-Length Locs. 5 and 6
Number of samples tested	33	21	17	42	22	22
Mean UTS (psi)	2,308	2,024	1,453	1,318	1,266	954
Median UTS (psi)	2,330	2,097	1,460	1,342	1,253	973
Standard Deviation (psi)	299	327	237	149	206	136
Coefficient of Variation	0.129	0.162	0.163	0.113	0.153	0.143
Weibull Modulus	11.6	6.3	6.7	10.1	6.8	7.7

^a Location numbers refer to Fig. 7.

The burst-strength values predicted by Eq. (7) are also shown in Fig. 12, together with values calculated while setting the volume-ratio term in Eq. (7) equal to 1. Fig. 12 shows that the observed strengths are higher than the ultimate tensile strengths obtained from the tensile samples, and also higher than the predicted strengths of Eq. (7). However, the values obtained by dropping the volume-ratio term from Eq. (7) are quite close to the observations.

Biaxial Tests on "Hole-Array" Samples

A number of graphite samples simulating a section surrounding a fuel hole in an HTGR fuel block were made from a block of H-327 graphite. The samples were tested by preloading in tension under loads of 0 to 1150 lb and internally pressurizing until the samples failed at a web. The geometry of the samples is complex, but since most of the load is borne by the band of solid material surrounding the central fuel hole it may be approximated to a thick-walled cylinder. The wall thickness of the equivalent cylinder was determined to be 0.23" by comparing the mean burst pressures under zero axial load with the burst pressures for the series of uniform cylinders described in the last section. For calculating the axial stress component supplied by the external load, the load was assumed to act over the full area of the cross section (1.625 sq in).

The maximum tangential stress at failure (calculated assuming elastic behavior) is plotted against the super-imposed tensile stress in Fig. 13. Also shown are the strengths calculated from Weibull theory and using the stress-ratio term alone [Eq. (10)]. As in the case of the regular tube-burst tests, the stresses calculated from the full equation including both a volume-ratio term and a stress-distribution term are considerably lower than the observed strengths. When the stress-distribution term alone is used, the calculated strengths agree well with the observations from samples taken near the outside edges of the block, but still fall somewhat low for the center and mid-radius positions.

DISCUSSION AND CONCLUSIONS

The distribution of strength values among a group of samples of H-327 graphite with the same orientation and log location is equally well described by a Weibull or a normal (Gaussian) distribution function (Figs. 8 and 9). Because the forms of the two distributions only differ significantly near the extremes of the strength ordinate, a large number of samples (> 100) would be needed to establish which distribution function fits H-327 better. However, the question may be of practical importance when the probability of failure at low stresses needs to be known (for example, in predicting the probability of failure in webs in PSC fuel blocks). As an example, for a Weibull modulus of 8 (mean value for H-327 graphite), a Weibull distribution of strengths would give a failure probability of 1.15% at 60% of the median fracture stress, whereas a normal distribution with the same standard deviation would predict a failure probability of only 0.3% at the same stress.

When enough tensile tests have been performed to obtain reliable values of the median tensile strength and the Weibull modulus, application of the Weibull theory makes it theoretically possible to predict the median strength of samples with other configurations and stress distributions. The ratio of the median strength of the samples with non-uniform stress systems, σ_{nu} , to the median tensile strength, σ_t , is always given by this type of expression:

$$\frac{\sigma_{nu}}{\sigma_t} = \left(\frac{V_t}{V_{nu}} \right)^{\frac{1}{m}} \times (\text{stress distribution term})$$

where V_t and V_{nu} are the volumes of the tensile samples and non-uniform stress samples respectively. The stress-distribution term depends on m and the way the stresses vary with position in the sample and the existence of biaxial stresses. Calculations of this term for various configurations are given in the Theory section of this report. The magnitude of this term is greatest for configurations where only a small part of the sample experiences the maximum stress, and for low values of m .

When the predicted strengths were compared with observed strengths for bend tests, tube-burst tests, and biaxial tube-burst tests on H-327 graphite (Application of Theory to Experimental Results section), it was found that the predicted values were too high in the case of the bend-tests (where the sample volume was lower than that of the tensile samples), and too low in the case of both sets of tube-burst tests (where the sample volumes were higher than the standard tensile samples). In addition, the bend-test results did not show the predicted dependence on sample width. However, when the volume-ratio term was placed equal to 1 and the stress-distribution term alone was used, the calculated values were much closer to the observed strengths, although the calculated values still tended to fall low for the tube-burst tests. One possible reason for this discrepancy is the deviation of the stress-strain curves from linearity which is a feature of all polycrystalline graphites. This tends to reduce the stress at the inner surface of a pressurized cylinder below the level calculated by an elastic analysis [13]. A similar effect occurs in a bend test, but to a much smaller degree.

It may be concluded that the changes in failure probability due to nonuniform stress distributions predicted by the Weibull theory are supported by the observations on H-327 graphite, but the expected dependence on sample volume is not observed. This lack of dependence on sample volume may occur because the Weibull derivation assumes that failure of the whole sample results from failure at the single worst flaw anywhere in the sample volume, whereas in practice localized cracking starts well before final fracture in graphite.

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Fig. 1. Probability Density Function vs Normalized Stress for a Weibull Distribution of Strengths and the Corresponding Normal Distribution. Weibull modulus, m , = 5 and 10.

Fig. 2. Ratio Between Median Modulus of Rupture and Median Ultimate Tensile Strength as Predicted by the Weibull Theory for Samples Whose Volumes Obey the Condition

$$V_t = V_{cs} + [2/(m + 1)]V_{os} ,$$

Where V_t is the Volume of a Tensile Sample, V_{cs} and V_{os} are the Volumes of the Center Span and Outer Span Respectively of a Bend Sample, and m is the Weibull Modulus.

Fig. 3. Strength of Internally Pressurized Thick-Walled Tubes Free from Axial Stress, as Predicted by Weibull Theory. The ratio of the median tube-burst strength to the median tensile strength of samples of equal volume is plotted as a function of (outside diameter/inside diameter).

Fig. 4. One-Half of the Tension-Tension Failure Envelope Predicted by the Weibull Theory for Failure Under Biaxial Stresses. For $m = \infty$, envelope becomes identical with a maximum principal stress failure criterion.

Fig. 5. Strength of Internally Pressurized Thick-Walled Tubes with Closed Ends (Axial Stress), as Predicted by Weibull Theory. The ratio of the median burst strength to the median tensile strength measured on samples of equal volume is plotted as a function of (outside diameter/inside diameter).

Fig. 6. Drawing of Sample Used for Biaxial "Hole-Array" Tests.

Fig. 7. Locations of Test Samples in a Log of H-327 Graphite.

Fig. 8. Cumulative Failure Probability vs Normalized Strength for Parallel Samples of H-327 Graphite (Edge of Block).

Fig. 9. Cumulative Failure Probability vs Normalized Strength for Perpendicular Samples of H-327 Graphite (Central Axis and Mid-Radius, Mid-Length of Block).

Fig. 10. Modulus of Rupture of Rectangular-Cross-Section Samples of H-327 Graphite Plotted Against the Mean UTS of Companion Samples. The theoretical relationships calculated from Weibull theory are shown.

Fig. 11. Modulus of Rupture of Cylindrical Flexural Samples Plotted Against the Mean UTS of Companion Samples. The theoretical relationships from Weibull theory are shown.

Fig. 12. Maximum Tangential Stress at Fracture for Tube-Burst Tests on H-327 Graphite, Plotted as a Function of (Outside Diameter/Inside Diameter). Values calculated from Weibull theory are also shown.

Fig. 13. Maximum Tangential Stress at Fracture for Biaxial "Hole-Array" Tests on H-327 Graphite, Plotted as a Function of Axial Stress at Fracture. Values calculated from Weibull theory are also shown.

THERMAL SHOCK AND ABLATION

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✓ THERMAL DISC TESTS FOR THE EVALUATION OF
GRAPHITE FOR AEROSPACE APPLICATION

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ABSTRACT

The thermal disc test was used to evaluate the thermal stress resistance of bulk graphites considered as candidate materials for aerospace applications. These graphites were evaluated on a relative basis by comparing the energy they absorbed without fracturing. The test samples were 1-1/4 in. in diameter by 1/10 in. thick. Thermal stress was introduced by heating the center of the disc with an electric arc on each face of the sample. More than 20 types of graphites from seven sources were evaluated.

Correlations were found between the energy to fracture and the grain size. Generally, finer grain structure sustained higher energy before fracturing. However, there is evidence that an optimum size of grain may exist: some of the finest structures had a fracture threshold below grades with slightly coarser structure. The influence of other physical properties is also discussed in terms of the sensitivity toward fracturing. The usefulness of this type of test as a screening device is reviewed.

The use of the thermal disc test for the evaluation of a number of different graphites is described and discussed, concentrating more on the structure of the graphite, rather than on the usefulness of the tests to predict effects under reentry conditions. The results have been used to point toward desirable changes in microstructure, hopefully improving the thermal stress resistance of graphites.

The test apparatus is shown in Fig. 1. The graphite disc is about 0.1 in. thick and 1.25 in. in diameter and is held in a clamp at the edge. Similarly machined surfaces are prepared for each of the specimens. The electrodes pour energy in from either side of the disc, and the power input is increased until fracturing of the sample occurs. Of course each

sample measures just one power level, because either the sample fractures or cavitation begins at the surfaces where the arc entered in the center of the disc.

With the assistance of the commercial producers of graphites, samples of different grades of graphite were obtained for comparative evaluation. These were subjected to different power levels, and the recorded results are shown in Fig. 2. The triangles are failures, and the circles are successes. As in many other investigations, at ORNL, IASL, and elsewhere, for each graphite a power level region was found in which successes and failures were mixed, a sort of gray zone. In some of the graphites, like the PO-3, as an example, there was a very small gray zone. Generally, the finer-grained graphites had smaller gray zones.

There was quite a spectrum in these various graphites, from 5, 6, or 8 kW all the way to some 30 kW. There were several experimental formulations in which the structures were changed intentionally to see what variations would occur, and apparently the microstructure does alter the susceptibility to thermal stress. Using this information, special formulations were made of the different microstructures.

The test became a device for ranking graphites according to thermal stress resistance, in order to begin to examine possible improvements in microstructure.

There have been a number of correlations and attempted correlations of thermal stress resistance with conductivity, strength, modulus, and expansion. However, we really do not quite know how these relate, especially under this dynamic situation and with these properties changing, and there may be other properties that are influential. The intent of this simplified test was to determine whether, in fact, there might be some other indications, and whether possibly these were influencing fracturing in ways that might help in the design of other microstructures.

A comparison of how the individual properties are changing with grain size is schematically depicted in Fig. 3. If they are combined, one might expect an optimum grain size. This is admittedly a simplified approach, but on the other hand the types of graphites produced in recent years are beginning to lend themselves to this type of approach. Graphites are made of more homogeneous materials now than in the days of larger grains and

wider variabilities. In the last ten years there has been an improvement in formulations, in what the customer can obtain in types of graphites which perform and are much more homogeneous in structure.

From some of the evaluations that have been made, it is believed that the grain size is a fairly important parameter from a pore volume point of view, as shown on the simple model of graphite structure in the paper by Meyer, Kratsch, and Dirling in the Material Characterization session of this conference. Figures 4 and 5 indicate that according to our estimate, the possible thickness of the binders around these grains is relatively small compared with some of the grain structures.

In Fig. 6 the circled graphites were either made in our laboratories or procured as special formulations. The uncircled graphites are commercial graphites. The ATJ-S is shown at about 154 microns, the special formulations at 60 to 80 microns, and POCO at about 15 microns. The results gave some feeling that there was a direction to move in preparing formulations for enhancing thermal stress resistance.

Most of the commercial graphites fell in one or the other end of the grain size spectrum. It seems fruitful to look between, and in fact an improvement was obtained in the thermal stress resistance. These tests do not show where the maximum power is, because the equipment ran out of power. It should be emphasized that these are all across-grain tests; many of these graphites did not fracture in with-grain tests.

From evaluations of these results, we think that there are other influences in addition to the physical properties that one normally measures individually. From a synergistic viewpoint there may be important factors such as grain size, or what it controls in terms of pore size and distribution.

Variations of some of these formulations were undertaken in one processing step or another, such as type of impregnant, type of heat treatment, or number of impregnations, in an attempt to alter the structure to see what it would mean in terms of the simple ranking order type of evaluation. There appears to be some influence. One formulation, with an additional impregnation, moved from 40 to 27 kW. Another formulation, with a higher heat treatment, moved from 50 to 37 kW. Thus, there are interrelated factors that we still have not characterized and mentioned.

The thermal disc test can be used, we believe, as an indicator of directions in which to move in microstructure, and we believe that there are in fact still directions in which to move. It is not sure whether the peak in Fig. 6 ought to be at 70 microns or somewhere over or under. As can be seen from the relative values, it can make a large difference in the amount of energy that can be put into a graphite.

In summary, there is still an ability for improving the thermal stress resistance of graphite. The simplified test is a relative indicator that can be used in these evaluations.

Fig. 1. Thermal Stress Disc Test with Instrumentation Removed.

Fig. 2. Thermal Stress Disc Data.

$$\sigma_R = \text{STRESS RESISTANCE} = f(\alpha, K, \sigma, E, A, B, \dots)$$

$$\sigma_R \approx \frac{K^x \sigma^y}{E a^z}$$

Fig. 3. Figure of Merit for Thermal Stress.

ATJ-S(S)	$\bar{L}(\mu)$	$\bar{t}_p(\mu)$
	70 (EST.)	20 (EST.)
MDAC-228	16	2

$$8t_B^3 + 2(3\bar{L} + 2t_p)t_B^2 + t_B(\bar{L}^2 + 3\bar{L}t_p)$$

$$= \left(\frac{K_B}{\rho_t} + v_{f_o} + v_{f_c} \right) \frac{\rho_t t_p \bar{L}^2}{2(1 - K_B)}$$

$$1 - \frac{S_c}{S} = \frac{3V_v d_v^2 \rho_t t_p \bar{L}^2}{(1 - K_B) d_v^3} \times$$

$$\left[\bar{L}^2 + 12\bar{L}t_B + 24t_B^2 + t_p(3\bar{L} + 8t_B) \right]^{-1}$$

Fig. 4. Flake Particle Model.

Fig. 5. Typical Binder Thickness for Graphite.

Fig. 6. Thermal Stress Resistance vs Maximum Filler Size and Processing Conditions.

X 2
A Temperature/Stress Test for Polygraphites

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ABSTRACT

A test has been developed that causes tensile fractures of the proper mode and at the proper location of test specimens made of polygraphites. A disc with a hole in the center is heated inductively at the outer periphery at rates to provide a temperature rise rate of the order of 2000°F/sec. Fracture occurs radially at the inner periphery during heatup at typically 2 to 4 seconds and may extend partially or totally through the section. In some cases, the disc may be wedged or rimmed at the outer periphery. A common geometry has been found that has fractured all polygraphites tried to date, including AXM, AXF, ATJ-S, ATJ and G-90. A rapid strain analyzer with an uncertainty of about 1/2 to 1 percent at any point on the scale is being developed to monitor the strains at the inner hole. Stress and deformation analyses of the disc are also being developed. When these two additional inputs are completed, studies of constitutive relations and failure criteria will be made.

As a corollary to the main effort, the partially fractured discs provide double-cantilever prenotched specimens for subsequent studies of fracture mechanics.

SYNOPSIS

The test consists basically of a disc of material which is heated inductively around the outer diameter so that tangential tensile stresses are produced around the inner diameter where failure

occurs. Figure 1 shows the three configurations which have been used in most of our work. The configuration shown in Figure 1a has been used more extensively so that a brief discussion on the operation of the test has been based on this specimen design. The specimen is a washer-like disc consisting of a thick outer rim and a thin central web. By heating the outer rim of this specimen rapidly and maintaining the central web relatively cool, failures can be generated in the inner web, usually in the form of one to three radial cracks.

The outer rim of the specimen is heated rapidly by inductive heating. A schematic of the setup is shown in Figure 2. The power supply is a Lepel high-frequency heating unit which is rated at 25 kW with an operating frequency of about 480 KHz. A 12:1 transformer is used in conjunction with the power supply. To minimize losses, the load length between the load coil and transformer is kept at a minimum.

A load coil of two turns has been employed in runs made to date. The copper tubing is 1/4 inch in diameter and is flattened in the area of the turns. The total height of the coil matches the thickness of the specimen rim. Good coupling with the specimen is assured by maintaining a small annulus of 1/16 to 1/32 of an inch between the outside diameter of the specimen and the inside diameter of the coil.

The specimen is supported and centered in the coil on a central post and cap assembly. The specimen is supported with this assembly so that it cannot turn or shift under the influence of the induced field.

With the specimen installed and the power supply preset at a selected position, the unit is turned on and the specimen heated rapidly. Since the induced power is concentrated in the outer rim of the specimen, the rim heats rapidly while the central web remains relatively cool. Under typical power input used to date, the rim

of the specimen reaches about 4000°F in 3-1/2 seconds while the central portion of the web remains below 1500°F. The expansion of the rim produces tensile stresses in the web and failures occur in the web.

One of the main problems with tests of this type is the determination of the actual power input into the specimen. Power readings can be made from the control console of the power supply; however, these readings will not necessarily reflect the true power input into the specimen. To date, we have been using a combination analytical-experimental technique to determine power input.

For a given material and power setting, the method is as follows:

Several specimens are heated and information for temperature-time and temperature-position are taken. This type of information for an ATJ graphite specimen (rimmed-disc) is shown in Figures 3 and 4. Here temperature measurements were made at four positions; each data point represents an average of three to five readings. To obtain the heat input, q''' , the calculated temperature profiles were fitted to the data points. (This is the same technique used in other high temperature analyses such as some thermal conductivity measurement systems and the better analyses developed for heat input to nose cones and rocket nozzles.)

At this point in time, the assumptions made in calculating the temperature profiles are:

1. All power is dissipated in the rim (the rim was nominally 1.75 times the "skin depth").

2. A radial temperature gradient is assumed with conduction in the radial direction and radiation losses from exposed surfaces. The value of the q''' (heat input per unit volume) is obtained by varying the q''' analytically until the calculated and experimentally measured temperatures agree as shown in the figures.

Figure 5 shows a plot of calculated power input versus power setting for four graphites. There are two significant features of this plot. One is the difference in the power inputs for the several graphites at a given power setting. The other is the apparent differences in the nature of the power input-power setting curves for the ATJ and G-90. In a relative ranking scale, a power setting of ~25 for ATJ gives nominally the same heat input as a power setting of ~50 for the G-90. This vividly points out the importance of knowing the actual test conditions as opposed to using knob controls.

Fractures have been generated in a variety of different graphites, some C-C materials and some graphite-carbide composites. Figures 6 through 11 show fractured specimens manufactured from ATJ, ATJ-S, AXM-5Q, G-90, ZTE and ZTA graphites. Note in all cases that there are generally two or more radial cracks. This is fairly typical. Most graphites remain intact; however, some, like the AXM-5Q shown in Figure 8, do fracture into several pieces.

Current plans are to develop this temperature/stress into an analytical tool that can be used to judge the applicability of current thermal stress analysis techniques and to suggest areas where more definitive materials' models or behaviors are required. To this end we have designed and are now building a rapid tracking extensometer. The extensometer will utilize four beams (two axes) of light focused on the edges of the hole in the center of the specimen. The beams will be servoed to track the specimen motions and measure dimensional changes that result from rapid heating in the induction furnace. Calculations and initial calibrations indicate that the extensometer will tolerate considerable specimen motion without exceeding 10 micro-inches of error for the 0.001 inch deformation. The minimum deformation that can be detected was estimated from theoretical considerations and appears to be less than 10 microinches.

To date the analytical aspects of the program have been limited to one-dimensional thermal analysis as noted earlier, and linear elastic (temperature dependent properties) stress analysis. Figures 12 and 13 show some typical results for ATJ-S graphite. As shown in earlier Figure 3, fairly good correlation has been obtained between some experimental temperature measurements and "analytical" predictions. Only limited measurements (time to fracture) have been available to try and correlate analytical stress predictions and there are no definite results beyond what is illustrated in Figure 13.

1a FLAT DISC

1b RIMMED DISC

1c WEDGE

Fig. 1. Disc-Shaped Specimens Used in the Temperature/Stress Test.

Fig. 2. Schematic of the Apparatus for the Temperature/Stress Test.

Fig. 3. Temperature vs Time at Several Radial Locations for an ATJ Temperature/Stress Specimen (RD).

Fig. 4. Temperature Profile at $t = 2.0$ sec for an ATJ Graphite Temperature/Stress Specimen (RD).

Fig. 5. Calculated Power Input vs Power Setting for Several Graphites.

Fig. 6. Temperature/Stress Disc, ATJ Graphite.

Fig. 7. Temperature/Stress Disc, ATJ-S Graphite.

Fig. 8. Temperature/Stress Disc, AXM-5Q Graphite.

Fig. 9. Temperature/Stress Disc, G-90 Graphite.

Fig. 10. Temperature/Stress Disc, ZTE Graphite.

Fig. 11. Temperature/Stress Disc, ZTA Graphite.

Fig. 12. Temperature Profile for ATJ-S Graphite Temperature/Stress Specimen (RD). $Q''' = 37$ Btu/sec-in.³.

Fig. 13. Temperature-Time and Stress-Time Plot for ATJ-S in the Temperature/Stress Test.

THERMAL SHOCK RESISTANCE OF EXPERIMENTAL GRAPHITES*

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ABSTRACT

We evaluated the potential of a number of experimental graphites for applications where thermal stress resistance is important. The test method for determining the relative thermal shock resistance of graphites consists of rapidly heating the center of a thin disk of graphite with an electric arc and maintaining a much lower temperature of the disk's periphery. The resulting radial temperature difference creates a tensile stress in the outer regions of the disk, and the disk will fracture if the stress is higher than the particular material will withstand. The rate at which heat is introduced at the center of the disk determines the temperature gradient and the magnitudes of the resulting stresses in the outer regions of the disk. The amount of power required to fracture a disk is a relative measure of the thermal shock resistance of the graphite being tested.

The test apparatus consists of a heat source, a support for the disk, a power supply, controls, and recording instruments. An inert-gas shielded nonconsumable-electrode arc welder is the heat source. A water-cooled copper plate with a hole in the center supports the graphite disk during the test. The arc variables such as cover gas composition and flow rate, electrode size, and distance from electrode to specimen are held constant so that the power introduced into heating the disk does not vary from one test to another. The total power expended by the welding machine is measured for each specimen, and the lowest power level which consistently produces fracture of an identical group of specimens is a measure of the material's resistance to thermal shock. This is referred to as the threshold power level and is proportional to the thermal shock resistance of the material.

We tested a series of developmental graphites fabricated at the Oak Ridge Y-12 Plant of Union Carbide Nuclear Division to determine the interrelated effects of various raw materials and manufacturing techniques on the thermal shock resistance of the finished material.

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A test method for determining the relative thermal shock resistance of graphite is described. This method has been used to evaluate the potential of a number of experimental graphites for applications where thermal stress resistance is important.

The thermal shock test, as performed at Oak Ridge National Laboratory, has been well documented. Several reports have been published describing the procedure used and the results obtained. The test has also been described at various meetings, including the Eighth and Ninth Conferences on Carbon. A brief description of the procedure used to obtain thermal shock resistance values of graphite follows.

A schematic block diagram of the test setup is shown in Fig. 1. A 300-amp DC welder is connected in straight polarity through a contactor and a high-frequency unit to the test stand. The high-frequency unit is used to initiate the arc. The straight polarity arrangement makes the specimen positive with respect to the electrode, imparting a maximum velocity to the electrons striking the graphite disk. The electrode holder is water cooled, and a cover gas of argon flows at 30 cfh around a thoriated tungsten electrode. The thermal shock specimen is placed over a hole in the copper plate. The plate is cooled by means of tap water circulating through a coil of tubing which is brazed to the underside. The periphery of the disk is thereby kept at a much lower temperature than the center of the disk, providing the thermal gradient necessary for the test. The system has been designed to test three sizes of specimens, each requiring a different support plate. Each specimen is normally tested only once. If the disk fails it cannot be tested again. If the disk does not fail, it is discarded and another disk is tested either at a higher or lower power setting. (A limited number of special disks were retested several times at progressively higher power levels, but this was a rare exception.) The largest disk is 4 in. in diameter by 0.2 in. thick, the smallest disk is $3/4$ in. in diameter by 0.05 in. thick, and the intermediate disk is 2 in. in diameter by 0.1 in. thick.

Each disk is finish ground flat and parallel to within 0.0002 in. total indicator runout. The plane of the disk is in the plane of isotropy of each of the particular grades of material used.

A photograph of a 2-in.-diam disk during a test is shown in Fig. 2. A steel ring is placed over the disk to keep it in contact with the copper plate. This eliminates secondary arcing between the disk and the plate. Some grades of graphite will fail catastrophically, while other grades have to be watched closely by the operator to determine whether and when they fail. In some instances a series of specimens will not fail at a particular power level, but will develop cracks upon cooling. Cracks which do develop during cooling are not considered as failures, and such specimens are tabulated as unbroken in the data log. These specimens must be watched closely during the test to avoid erroneous results.

For some tests a crack detector is employed. The probe of the crack detector is visible in the upper portion of Fig. 2. The detector is an electromechanical transducer which contacts the edge of the disk and produces a signal when a specimen fails. The crack detector functions most satisfactorily on material that fails catastrophically.

Figure 3 is a photograph of a 3/4-in.-diam disk undergoing test. These 3/4-in.-diam disks are tested with the electrode holder positioned above the specimen. For this test a vacuum line is attached to the bottom of the plate. Vacuum is used in lieu of the steel ring to hold the specimen in contact with the plate.

The three different sizes of disks are shown in Fig. 4. The AGOT disks on the left failed but remained in one piece. The RVD disks on the right failed catastrophically, which is most often the case. The disks fail in two and occasionally three pieces.

During the test the welder current and voltage output are continually recorded, as shown in Fig. 5. The chart travels in the downward direction. The current and voltage remain fairly constant after the arc has

once stabilized. The blips are caused by a voltage surge when the specimen failed, and not from the crack detector described previously. In this figure, specimen No. 2674 is the only one that did not break. After approximately 22 seconds the arc was terminated and disk 2674 was removed from the plate and discarded.

After a series of disks from a particular material are tested, the data are tabulated as shown in Table 1. The specimens are listed in order of increasing power. The run numbers indicate the order in which the specimens were tested. The welder setting is the nominal current at which the welder was set. The time is the duration of the test. The last column indicates whether the disk failed. The lowest power at which a disk fractured was 3537 watts, and the highest power at which a disk did not fracture was 5540 watts. The bar graph shown in Fig. 6 depicts the thermal rupture resistance of some commercial grades of graphite. The plane of the disks for the extruded material (viz: British Isotropic, G-5, Graphitite "G", SX-5, 780-S, and AGOT) was in the across-grain direction. The rest of the graphites were molded, except ZTA. The plane of the disks for the molded grades was in the with-grain direction. This would account for the fact that the thermal shock resistance of the molded material is generally higher than that of the extruded graphites. The thermal shock resistance of the hot-pressed, highly anisotropic, Union Carbide grade ZTA was beyond the capacity of the welder for the 2-in.-diam disks.

The dotted portion of the bar is the power level range in which no specimens failed. The right-hand end of the bar is the power level at which all specimens failed. The range of power within the crosshatched portion of the bar is where some disks failed and some did not. It is within this range that the threshold power level falls. The threshold power level, defined as the lowest level at which disks of a particular graphite consistently fracture, is a measure of the thermal shock resistance of the material.

The foregoing procedure is an excellent method of screening various graphite materials where thermal shock is important. More than 4500 specimens comprising 150 different grades have been tested using this method.

For a particular structural application it was desired to make a material which possessed a high coefficient of thermal expansion plus a high resistance to thermal shock. Two types of fillers and binders were used to make a series of eight experimental graphites. The fillers were POCO AXM and Santa Maria coke. The binders used were Varcum (a glassy carbon) and Isotraxene, a thermoplastic graphitizable resin. These fillers and binders were combined in four groups, and each group was molded, fired, and graphitized into material whose thermal shock resistance was determined.

Table 2 lists these graphites along with some of their physical properties and their thermal shock threshold power levels. These are all reconstituted, molded graphites with the exception of the Great Lakes GL-1008 which is included for reference as a standard of comparison. The four combinations of filler and binder are: Santa Maria with Varcum; Santa Maria with Isotraxene; POCO AXM with Varcum; and POCO AXM with Isotraxene. All combinations of filler and binder received a final graphitization at 2800°C. All the fillers contained 15% Thermax. The particle size distribution for the Santa Maria filler is designated by F (fine), M (medium), and C (coarse). The particle size has very little effect on the coefficient of thermal expansion, but the thermal shock resistance is slightly higher for the graphite made from coarse Santa Maria filler. Comparing the Varcum bound AXM and Santa Maria F (which have similar particle size distributions) at the same molding pressure (5000 psi) shows the AXM to have both a higher coefficient of thermal expansion and higher thermal shock resistance. Increasing the molding pressure on the AXM bound with Varcum slightly increases its coefficient of thermal expansion and its thermal shock resistance.

The graphite made from Santa Maria F filler and Isotruxene binder exhibits the best combination of a high coefficient of thermal expansion plus a high resistance to thermal shock. It has the same thermal shock resistance as the reference GL-1008 No. 4 graphite and the highest coefficient of thermal expansion of all the graphites. The coefficient of thermal expansion of the graphite made from Santa Maria F filler and Isotruxene binder is 186% of the coefficient of thermal expansion of the reference GL-1008 No. 4 graphite.

Table 1. Thermal Shock Data for 2-in. PO3 Graphite Specimens

Run Number	Welder Setting	Current (amps)	EMF (volts)	Power (watts)	Time (sec)	Broke
1438	100	108	26.0	2808	16.0	No
1453	125	132	26.1	3445	15.0	No
1454	125	133	26.1	3471	16.0	No
1451	130	135	26.1	3526	16.0	No
1441	135	140	25.2	3528	15.0	No
1440	125	135	26.2	3537	15.0	No
1450	130	135	26.2	3537	15.0	No
1447	130	135	26.2	3537	15.0	No
1452	130	135	26.2	3537	8.0	Yes
1449	130	135	26.3	3550	15.0	No
1448	130	135	26.3	3550	5.0	Yes
1455	135	141	26.0	3666	16.0	No
1442	140	145	25.8	3741	15.0	No
1446	135	140	27.0	3780	3.5	Yes
1456	140	145	26.1	3785	16.0	No
1445	140	145	26.7	3872	6.0	Yes
1443	145	148	26.7	3952	4.0	Yes
1444	145	148	27.0	3996	6.0	Yes
1457	145	153	26.2	4009	15.0	No
1458	150	160	26.9	4304	15.5	No
1462	150	160	26.9	4304	15.0	No
1439	150	165	26.8	4422	4.0	Yes
1463	155	165	26.9	4439	15.0	No
1460	155	165	27.0	4455	3.5	Yes
1461	155	173	25.9	4481	3.0	Yes
1459	155	165	27.6	4554	5.0	Yes
1464	160	173	27.0	4671	15.0	No
1465	165	182	27.1	4932	15.0	No
1466	170	185	27.9	5162	3.0	Yes
1468	170	185	28.0	5180	15.0	No
1467	170	185	28.9	5347	3.0	Yes
1437	175	205	27.0	5335	3.0	Yes
1471	175	200	27.7	5540	15.0	No
1470	175	198	28.0	5544	5.0	Yes
1469	175	199	27.9	5552	5.0	Yes
1472	175	200	28.0	5600	5.0	Yes
1436	225	255	27.0	6885	2.0	Yes
1435	275	310	29.2	9052	2.0	Yes

Table 2. Data For Y-12 Molded Experimental Graphites

Filler ^a	Filler Firing Temperature (°C)	Binder ^b	Molding Pressure (psi)	Bulk Density	CTE _{WG} ^c 10 ⁻⁶ in/in/°C (30 to 1000°C)	Threshold Power (watts)
GL-1008 #4	2300	Varcum (22)		1.72	3.6	2200
Santa Maria F	2500	Varcum (23)	5000	1.72	5.3	900
Santa Maria M	2500	Varcum (23)	5000	1.56	5.2	850
Santa Maria C	2500	Varcum (23)	5000	1.58	5.4	1550
POCO AXM-1	2300	Varcum (27)	2500	1.64	5.4	900
POCO AXM-2	2300	Varcum (27)	5000	1.66	6.6	1250
Santa Maria F	2500	ITX (23)	2400	1.78	6.7	2200
Santa Maria C	2500	ITX (23)	2400	1.57	4.7	2100
POCO AXM	2300	ITX (27)	2400	1.74	5.0	2300

^aAll fillers contained 15% Thermax.

^bNumbers in parentheses indicate parts of binder per hundred parts of filler.

^cCoefficient of thermal expansion in the with-grain direction.

Fig. 1. Schematic of Power Circuit for Thermal Shock Test.

Fig. 2. Graphite Disc, 2 in. in Diameter, During a Test.

Fig. 3. Test Rig for 3/4-in.-Diam Specimens.

Fig. 4. Broken Thermal Shock Specimens.

Fig. 5. Typical Brown Recorder Chart from Thermal Rupture Tests.

Fig. 6. Thermal Rupture Resistance of 2-in.-Diam Specimens.

✓ CORRELATION OF EXPERIMENTAL CARBON AND
GRAPHITE ABLATION DATA

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ABSTRACT

Graphite and carbon serve as thermal protective materials on reentry vehicles by ablating slowly at very high temperatures. To evaluate and compare ablation of various materials in several test facilities, a relationship is presented in which recession rate is related to stagnation pressure and gas enthalpy. The proportionality constant acts as the basis for the comparisons. It was found that material and testing variabilities permitted little or no differentiation in the ablation performance of the seven bulk graphites and eight modifications of them. Five of the six test facilities used gave similar results. These results suggest that factors other than recession rate should be used as a basis for selecting a bulk graphite.

Introduction

Polycrystalline or bulk graphites are being used as replacements for resin composites in nosetips and heatshields for reentry vehicle applications. More recently, graphite composites have also entered the field as candidates for these applications. These graphitic materials are of particular interest because they have a high sublimation temperature, excellent high temperature strength to weight ratios, good ablation resistance and acceptable shock resistance.

Recent, more severe reentry requirements have placed even greater demands on graphitic materials and accentuated the needs for a better understanding of the ablation process. These needs relate to a capability for predicting ablation performance over a broad spectrum of environmental conditions so that appropriate materials and designs can be implemented. Present

predictions for the response of these materials to reentry conditions come from computer codes and ground test results in high temperature jets. Attempts to formulate computer codes which take into account known mass removal mechanisms (chemical, physical and mechanical) in a material which is under thermal and mechanical stress have provided some success for predicting ablation under mild reentry conditions. Under more severe conditions these codes are considerably less accurate. An additional drawback of these codes is the absence of any provision for differentiating between the various graphites available for reentry applications.

Ground testing as a source of information for the ablation process also leaves much to be desired. First, the pressure and enthalpy conditions which may be obtained in ground test facilities do not include all conditions experienced in flight. Secondly, the spectrum of conditions covered by each facility is limited such that a number of facilities must be used to evaluate and rank the materials under study as well as broaden our understanding of the ablation process. This necessitates additional testing, thereby entailing additional expense.

In the absence of sophisticated knowledge of material properties and ablation processes, it becomes desirable to have a simple, empirical, albeit approximate, relationship which can be used to predict ablation and rank materials. Rankings would be made in terms of a parameter which would normalize test conditions.

A relationship of this type is given below.

$$\dot{s} = A_s P_m H_o^2 \quad (1)$$

where

$\dot{s}$ = recession rate, in/sec

A_s = ablation parameter, in-lb²/sec-atm-BTU²

P_m = model stagnation or impact pressure, atm

H_o = bulk gas enthalpy, BTU/lb

This report provides evidence for this relationship and compares the values obtained for A_s from different materials and test facilities. It shows that the A_s values possess broad scatter bands which do not permit differentiation in ablation performance for most test facilities and materials investigated. This conclusion is of particular importance since much effort in ablation studies is concerned with material ranking.

Materials, Test Facilities, Test Conditions and Model Geometries

The graphites and carbons in Table I provided the ablation data from which the conclusions of this report were derived. Six test facilities were used (Table 2). They provided a broad range of stagnation or impact pressures

and gas enthalpies. Details concerning testing and test conditions are reported in References 1-6. Two model geometries were used; hemisphere cylinders were tested at AEDC, AVCO, MDAC, Sandia and Plasmadyne. Cone cylinders were tested at CAL and MDAC. Schematics of the AEDC and CAL models are shown in Figures 1 and 2. The diameters of the hemisphere cylinders were 0.4 inch for models tested at AEDC, AVCO and Plasmadyne, and 0.3 inch for models tested at MDAC and Sandia. The included angle of the cones was 130° and the cylinder diameter was 0.5 inch.

Recession Rate Measurements

Ablation was measured in terms of the linear recession along the axis of the cylinder. High-speed motion cameras recorded the ablation and a film reader provided recession data as a function of time. A computer program converted the data into smoothed plots from which plots of the recession rate ($\dot{s}$) vs time were obtained. This method of data reduction was used for the AEDC, AVCO and CAL data. The MDAC data, however, had not been reduced at the time this report was written. Only preliminary recession data are given therefore for MDAC test results in this report.

Most of the recession vs time plots approached linearity especially during the initial stages following temperature equilibration. This will be referred to as the quasi-steady-state regime. Deviations from linearity could usually be attributed to massive spallation or retraction of the model from constant environmental conditions.

Three types of recession rates were used to evaluate the ablation parameter A_s : (1) An average or gross recession rate, obtained from the total recession and the test time; (2) an initial rate, obtained by extrapolating the rate in the quasi-steady-state regime to zero time; and (3) an average initial rate obtained over the quasi-steady-state before deviations from this state appeared.

The standard deviation for A_s was used to determine which of the above methods gave the most consistent results. Analyses of the AEDC and CAL results showed that Method (3) gave A_s values with the smallest standard deviations whereas Method (1) gave the largest standard deviations. Method (3) was therefore used for all tests when sufficient data were available.

Results and Discussion

This report provides evidence for Eq. (1) and the applicability of A_s as a basis for comparing materials and test facilities. Validity for Eq. (1) was established by plotting recession rates as functions of pressure and the square of the enthalpy. The recession rates were normalized for enthalpy and pressure when these conditions varied within a test series.

Figure 3 shows the linear relationship between recession rate and pressure for tests conducted at MDAC with ATJ-S and IP-50. The enthalpy was essentially constant in this series. The two lines describe a scatter band for the data. Figure 4 shows a similar plot for ATJ-S tests conducted at CAL, AEDC and Sandia (lower line). Because the enthalpies were

not constant in tests whose results are plotted in Figure 4, the recession rates are normalized. The Sandia and AEDC data describe a linear relationship which appears to be supported by the CAL data although scatter of the CAL data is quite large. The MDAC data are also included in this figure. It is obvious that the latter data describe a different linear relationship. This difference will be discussed below.

Figure 5 shows the effect of enthalpy on recession rate (normalized for pressure) for ATJ-S. Here again the Sandia and AEDC data define a linear relationship which appears to be supported by the CAL data. The MDAC point which is a mean value for all MDAC-ATJ-S data is obviously not on this line.

Better evidence for the linear relationship between recession rate and the square of the enthalpy is obtained from test data with AXF-5Q graphite. This material provided more consistent data and, since it was included in the AVCO test series, a greater range of enthalpy values was available. These data are shown in Figure 6.

Having established that Eq. (1) describes the data and data trends over the range of test conditions covered in this report, it was of interest to compare materials and test facilities in terms of A_g . These values were obtained from individual tests using the respective recession rates, pressures and enthalpies.

The A_g values were analyzed statistically to determine whether significant differences existed in these values. An F-test was used when possible to check for equality of variances and a student t-test was used to check for equality of means. If both tests gave nonsignificant results (95% confidence level), the materials were judged to be statistically the same. Because of the limited data in some cases, the tests were not very powerful, i.e. the probability of detecting material differences when differences existed was not very high.

The AEDC Test Series III provided an opportunity to compare ATJ-S with several IP graphites. The latter materials consisted of short fiber (2 x 250 μ) composites in phenolic and/or pitch matrices which had been heat-treated to 2500-3000°C. The A_g values which are given in Table 3 are particularly instructive because they provide measures of (1) variability within tests, in which a maximum of five models were exposed, (2) variability between tests, and (3) material variability. It is evident that Tests 8, 9, 12 and 13 constitute one group in which there are no statistically significant differences between values of A_g and their standard deviations. Similarly, Tests 10 and 11 constitute a consistent group with results significantly different from the others. It should be noted that the reported test conditions for Tests 10 and 11 were approximately the same as those for Tests 9 and 12, respectively, indicating that the variation may have been due to questionable diagnostics of test conditions rather than material variability.

To obtain a reasonable sampling for the above analyses, the results for ATJ-S and IP have been combined. Therefore, the above conclusions are based on the assumption that no significant difference exists between ATJ-S and IP graphites. Evidence for this assumption is given in Table 4 where it is seen that variability between these materials is not significant.

The data for the test series conducted at CAL are shown in Table 5. The variabilities in the CAL tests are greater than those observed at AEDC and are sufficiently large that any existing differences between materials tested at CAL and the same materials tested at AEDC (Tables 4 and 7) are masked.

The MDAC tests were conducted with ATJ-S and IP-50 models. Although the A_s values obtained from these tests are preliminary (Table 6), they are instructive when compared with those from AEDC and CAL. They contrast with those obtained at AEDC in two respects: (1) They are significantly larger; and (2) IP-50 is significantly different from ATJ-S, whereas the IP and ATJ-S results from AEDC do not differ.

The differences in the AEDC and MDAC results may be due to different flow conditions in the plasma around the models; i.e. laminar at AEDC and turbulent at MDAC. This suggestion stems from the observed model shapes following testing. At AEDC, the hemisphere cylinders were converted to right- and elliptically-tipped cylinders; whereas, at MDAC they were converted to cones. These shape changes are generally associated with the above flow conditions.

Flow conditions at CAL were also turbulent. However, the mean A_s value for ATJ-S, although larger than that from AEDC, as expected, is also smaller than that from MDAC. Unfortunately, the variability at CAL is much greater, and therefore does not permit a distinction between A_s values from this facility and those from AEDC and MDAC.

The difference between IP-50 and ATJ-S observed at MDAC and the absence of a corresponding significant difference in the AEDC data suggest that the ablation resistance of IP-50 in terms of recession is less than that for the IP graphites listed in Table 4.

Both recession and mass loss rates are bases for evaluating ablation performance and the two should be related by density. However, such factors as shape change and irregular mass removal can cause significant differences in apparent ablation as measured by these criteria. Since ablation is a surface phenomenon, it is dependent on the mass per unit area of surface. Thus, the density becomes an important factor in the characterization of ablation performance. This subject is relevant to the present discussion because the difference between the A_s values for ATJ-S and IP-50 in Table 6, although significant, is marginal. Therefore, the densities of the two materials should also be considered.

The densities of ATJ-S and IP-50 are 1.82 and 1.69 g/cc, respectively. When the A_s data in Table 6 are corrected for density, the difference does indeed become insignificant, showing that the observed difference in recession rates is due in part to a density variation. Density differences did not affect the conclusions derived for other test series or materials in this report.

Earlier ablation testing conducted at AEDC and AVCO provided A_s values for a broader range of materials. These are listed in Table 7. With the exceptions of CVD felt and AXF-5Q, the samplings are small and do not permit

narrow confidence intervals. The data show that CVD-felt is different* from the other materials and that significant differences do not exist for the other materials within each facility nor between the facilities.

Table 8 shows the A_s values for AXF and ATJ-S obtained at the Sandia and Plasmadyne facilities. Gross recession rates, rather than initial rates, were used to compute A_s . These values are consistent with those obtained at AEDC, AVCO and CAL.

In summary, the ablation studies of seven materials and eight modifications which were tested at six facilities over a broad range of conditions show that material and testing variabilities permit little or no distinction among the bulk graphites. CVD-felt, which is not a bulk graphite, is markedly different. Of the six test facilities used, only MDAC provided significantly different values for A_s . It is concluded that in the absence of more uniform graphites and better test controls, the selection of a bulk graphite for aerospace applications should be based on criteria other than ablation performance.

Conclusions

A relationship which involves recession rate as a function of stagnation pressure and gas enthalpy is valid for the test conditions studied. The proportionality constant in this relationship is used to compare the ablation resistance of seven graphites and carbons as well as eight modifications of these materials in six plasma facilities under a broad range of test conditions. Material and test variabilities did not permit distinctions between the ablation resistance of the bulk graphites. The test results from AEDC, AVCO, Sandia and Plasmadyne were indistinguishable. Those from MDAC, however, were different from the above. Material and test variability did not permit a distinction between the results from CAL and the other facilities.

Acknowledgment

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*Preliminary results from more recent tests in a channel test device with improved CVD-felt materials show it to be superior to the bulk graphites.

Table 1. Graphites and Carbons Studied

Material	Test Facilities*	Supplier	Description
ATJ	AEDC AVCO	Union Carbide Corp.	Medium grain molded graphite, graphitized at 2500°C
ATJ-S	AEDC CAL MDAC SIA	Union Carbide Corp.	Medium grain molded graphite which is of higher purity and density than ATJ; graphitized at 2800°C
AXF	PLAS	POCO Graphite, Inc.	Fine grained, isotropic graphite; graphitized at 2300°C
AXF-5Q	AEDC AVCO CAL	POCO Graphite, Inc.	Fine grained, isotropic graphite; graphitized at 2500°C
AXF-5Q (densified - not annealed)	AEDC AVCO	POCO Graphite, Inc., modified at Battelle	Hot-isostatically compacted at 2650°C and 13,000 psi
AXF-5Q (densified and annealed)	AEDC AVCO	POCO Graphite, Inc., modified at Battelle	Modified as above and annealed
ACF-4Q	AEDC AVCO	POCO Graphite, Inc.	Fine grained carbon; heat-treated at 1400°C
Graph-i-tite G	AEDC AVCO CAL	Carborundum Co.	Large grained extruded graphite; graphitized above 2500°C, highly anisotropic
IP	AEDC MDAC	AEC Y-12 Plant	Fiber (2 x 250μ) - pitch and/or phenolic resin-composite; graphitized at 2500-3000°C
P-03	AEDC AVCO	Pure Carbon Co.	Fine grained molded graphite; graphitized above 2500°C
CVD-felt	AEDC	Super Temp Corp. and SIA	Chemical vapor deposition of carbon on felt. Deposition temperatures 1200-1300°C. Some samples were hot pressed at 2600-3000°C.

* AEDC - Arnold Engineering Development Center

AVCO - AVCO Space Systems Division

CAL - Cornell Aeronautical Laboratory, Inc.

MDAC - McDonnell-Douglas Aircraft Corp., St. Louis, Mo. Division

SIA - Sandia Laboratories, Albuquerque, New Mexico Division

PLAS - Plasmadyne - Division of Geotel, Inc.

Table 2. Test Conditions

Facility	Model Stagnation Pressure, P_m (atm)	Bulk Gas Enthalpy H_o^{298} (BTU/lb)
AEDC 5 MW Heater	35	3330
	45	3335
	52-57	2210-2700
	76	2215-2395
AVCO 10 MW Arc Facility	6.7	6300-6700
CAL WAVE Superheater	58	1590
	63-64	1560-1590
	73	1660
	72-76	2000-2110
	83-84	1580-1630
	91	1340
	101-103	1240-1250
	101-110	2020-2550
MDAC-HIP 12 MW High Pressure Arc Heater	51-165	2050-2200
Sandia Laboratories 1.25 MW Arc Jet	13.1	3104-3187
Plasmadyne 1 MW Arc Jet	6.0	3000

Table 3. Comparative A_s Values from Tests Involving Groups of
ATJ-S and IP Graphites in Test Series III Performed at AEDC

<u>Test</u>	<u>Material</u>	<u>No. of Models</u>	<u>Stagnation Pressure, atm</u>	<u>Gas Enthalpy, BTU/lb</u>	<u>Mean A_s^a</u>	<u>Standard Deviation $\times 10^{10}$</u>
8	ATJ-S IP	2 3	35	3330	2.32	0.20
9	ATJ-S IP	2 3	56	2340	2.14	0.21
10	IP	5	56	2355	1.69	0.25
11	ATJ-S IP	1 2	76	2215	1.73	0.24
12	ATJ-S IP	1 3	76	2395	2.47	0.20
13	ATJ-S IP	2 2	45	3335	2.60	0.20

$a(\text{in-lb}^2/\text{sec-atm-BTU}^2) \times 10^{10}$

Table 4. Comparative A_s Values for ATJ-S and
IP Graphites from Test Series III at AEDC

<u>Material</u>	<u>No. of Models Tested</u>	<u>Mean A_s $\frac{\text{in-lb}^2}{\text{sec-atm-BTU}^2}$ $\times 10^{10}$</u>	<u>Standard Deviation $\times 10^{10}$</u>
ATJ-S	7	2.29	0.21
IP 22E	3	2.40	0.17
IP 22F	3	2.58	0.24
IP 22C	2	2.60	0.27
IP 17	3	2.14	0.30
IP (Mean)	11	2.41	0.28

Table 5. Comparative A_s Values for
Materials Tested at CAL

<u>Material</u>	<u>No. of Models</u>	Mean A_s	Standard Deviation $\times 10^{10}$
		$\frac{\text{in-lb}^2}{\text{sec-atm-BTU}^2}$ $\times 10^{10}$	
ATJ-S	9	2.87	1.11
Graphitite G	5	3.10	1.42
AXF-5Q	8	2.52	0.97

Table 6. Comparative A_s Values for Tests
Conducted at MDAC with ATJ-S and IP-50

<u>Material</u>	<u>No. of Models Tested</u>	Mean A_s	Standard Deviation $\times 10^{10}$
		$\frac{\text{in-lb}^2}{\text{sec-atm-BTU}^2}$ $\times 10^{10}$	
ATJ-S	10	4.08	0.18
IP-50	8	4.51	0.31

Table 7. Comparative A_s Values for Graphites and Carbons

Tested at AEDC (Series I and II) and AVCO

Material	Sampling		Mean A_s and Standard Deviation	
	AEDC	AVCO	AEDC	AVCO
ATJ	2	2	$A_s = 3.61 \times 10^{-10}$ $s = .64$	2.07×10^{-10} .11
Graphitite-G	2	1	$A_s = 2.02$ $s = .18$	.95 0
ACF-4Q	2	2	$A_s = 2.93$ $s = 1.03$	1.42 .66
AXF-5Q (as received)	6	2	$A_s = 2.22$ $s = .68$	2.37 .28
AXF-5Q (compacted to 2.13 g/cc and unannealed)	1	2	$A_s = 1.78$ $s = 0$	2.36 .16
AXF-5Q (compacted and annealed)	3	0	$A_s = 1.96$ $s = .64$	
P-03	2	2	$A_s = 2.68$ $s = .85$	2.15 .31
CVD-Felt	7	0	$A_s = 6.30$ $s = 1.68$	

Table 8. Supplementary A_s Values from the
Sandia and Plasmadyne Test Facilities

<u>Material</u>	<u>Test Facility</u>	<u>No. of Models Tested</u>	Mean A_s
			$\frac{\text{in-lb}^2}{\text{sec-atm-BTU}^2} \times 10^{10}$
ATJ-S	Sandia	1	2.43
AXF	Plasmadyne	1	2.00

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Fig. 1. Schematic of the Ablation Model Used at AEDC. The Model Used at MDAC is Similar in Geometry but with a 0.3-inch Diameter.

Fig. 2. Schematic of the Ablation Model Used at CAL.

Fig. 3. Effect of Pressure on the Recession Rate of ATJ-S Graphite.

Fig. 4. Effect of Pressure on the Recession Rate (Normalized for Enthalpy) of ATJ-S Graphite.

Fig. 5. Effect of Enthalpy on the Recession Rate (Normalized for Pressure) of ATJ-S Graphite.

Fig. 6. Effect of Enthalpy on the Recession Rate (Normalized for Pressure) of AXF-5Q Graphite.

STRUCTURAL DESIGN CRITERIA AND PROOF TESTING

Chairman: *G. B. Marrow*, Oak Ridge Y-12 Plant

X NERVA DESIGN CRITERIA AND APPLICATION FOR BRITTLE MATERIALS

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ABSTRACT

Brittle materials have become of more and more interest for structural use in recent years. Their structural adequacy at high temperatures makes them particularly well suited for various aerospace and nuclear applications. However, the accompanying low ductility and high sensitivity to crack propagation require that conservative design criteria be applied when using these materials for structural members.

This paper describes the SNPO NERVA method of analysis for brittle materials with particular emphasis on fracture mechanics and Weibull techniques. Since there is no well defined separation between brittle and ductile materials, a method of classification to be used in conjunction with this analysis is also presented. This method considers not only the type of material but also its structural application, the environment to which it is subjected, and its sensitivity to crack propagation.

A specific example of this design methodology coupled with modern probabilistic techniques leading to high reliability in the initial design is presented.

NOTE: The Nuclear Engine for Rocket Vehicle Application Program (NERVA) is administered by the Space Nuclear Propulsion Office (SNPO), a joint office of the U. S. Atomic Energy Commission and the National Aeronautics and Space Administration. Aerojet Nuclear Systems Company as prime contractor for the engine system and Westinghouse Electric Corporation as subcontractor for the nuclear subsystem, are developing a nuclear propulsion system for space applications.

INTRODUCTION

The NERVA program is using a probabilistic approach to design analysis, rather than a deterministic approach with safety factors used to account for uncertainty. The failure mode analysis is the principle analytical tool used in the probabilistic analysis design approach. It is a design approach that lists the possible ways a component or part ceases to perform an intended function, the mechanism that could cause the failure, and the analysis to determine the probability of recurrence of the failure modes.¹

Structural engineers in almost all fields of mechanical design have for years used safety factors based on experience in design. By a trial and error method a particular value of safety factor was determined through operational experience. This safety factor became the factor used in design and was accepted as adequate and used universally for similar applications. The method has worked out fairly well and many complicated designs have operated safely based on very convincing operational experiences. This deterministic approach, however, is being supplanted in many fields by probabilistic procedures which approximate reality better and permit an analytical optimization between cost and weight savings and the probability of a component fulfilling its intended purpose.

Since the methodology and thorough engineering understanding needed to apply these more advanced probabilistic methods is only partially available, there has to be a period of time where prior safety factor design type practice has to be used along with the application of these newer probabilistic procedures. NERVA is no exception to this and the Space Nuclear Propulsion Office has issued a controlling structural design standard which establishes the approach to be used in this interim period.²

In the current NERVA preliminary design phase the NERVA design groups will start applying the probabilistic approach with what amounts to immature data side-by-side with more conventional approaches. As the analysis, materials, test and development program unfolds this data will be matured such that the probabilistic approach will become the primary basis for establishing the NERVA design. By the time the qualification stages are reached for the various components, subsystems, and finally the engine itself, this approach should have been fully matured in all respects.

In designing for high reliability it is not only necessary that the structural engineer meet structural and probabilistic requirements but he must also insure that the method of analysis is in agreement with the material classification. Unconservative results can readily occur by applying methods of ductile analysis to brittle materials. Such may be the case when analyzing a structure made from a normally ductile material containing critical flaws. The situation may be further aggravated if the structure is subjected to cyclic loading or hostile environments.

A method for defining a brittle behavior state, based on fracture mechanics, is described in this paper. Following this classification procedure, methods of analysis for brittle materials, including Weibull techniques, are discussed. Finally, margin of safety and reliability procedures are defined.

FLAW SENSITIVITY CLASSIFICATION

All materials included in the NERVA program shall be classified as ductile or brittle with respect to a particular application and operating environment. Quasi-brittle materials, that is, materials exhibiting brittle tendencies due to the presence of an identifiable flaw under certain structural applications, will be initially classified as brittle. This conservative ductile-brittle classification shall be done by a committee consisting of materials, inspection and structures groups. The classification shall be based on all available materials properties data and past experience, and shall include estimates of flaw characteristics and critical stress intensity factors.

A material shall be considered to be in the brittle state if it demonstrates a sensitivity to flaws. Flaw sensitivity is exhibited if, for an arbitrarily defined flaw size, a_m , and the initial critical stress intensity factor, K_i , the critical stress intensity is less than or equal to the material yield strength. The basic equation used for determining the critical stress intensity, σ_c , is

$$\sigma_c = \frac{K_i}{Q \sqrt{\pi a_m}} \quad (1)$$

This equation is sketched in Figure 1 and the terms used explained in the following paragraphs.

K_i - The Initial Critical Stress Intensity Factor

The commonly reported material fracture toughness value, K_{IC} , is defined as the critical stress intensity factor at which unstable crack propagation occurs. This value is obtained from plane strain specimens loaded in the opening mode in accordance with ASTM accepted practice. Oftentimes a structure is not in a state of plane strain and should be described by K_C values. Since no standard procedures have been defined for K_C testing it is necessary to perform full or reduced size component tests when other than plane strain situations prevail.

In actual service it has also been observed that significant slow crack propagation may take place in structures subjected to fatigue loading or sustained loading in hostile environments. In these cases it is possible for sub-critical crack sizes to slowly grow to a critical size where rapid propagation ensues. This time dependent crack growth can be accounted for by basing the initial allowable crack size or stress on a reduced K_C value. This reduced value is defined by the symbol K_i . (Note that K_i for static loading, plane strain situations can also be denoted by K_{IC} .)

For fatigue loadings K_i is a function of the number of expected cycles and the range of applied stress intensity for each cycle. For sustained loading in hostile environments (also called stress-corrosion cracking) K_i depends upon the magnitude of the load, the time under loading and the environment. Figure 2 shows typical reduced K_C values due to fatigue and sustained loadings.

For many materials there is not necessarily a lower limit to the fatigue degrading effect, whereas for sustained loading there is usually a very definite lower limit. This lower limit is defined as the threshold K_I of K_{TH} . In cases where both fatigue and hostile environments are present the degrading effects must be combined in the experimental design as well as in the analysis.

a_m - The Classification Flaw Size

Flaw sizes greater than or equal to the transition value, a_T (see Figure 1), can cause unstable crack propagation in a structural member at stresses lower than the yield strength. Such flaws may be introduced during fabrication or machining and are difficult to control. However, reliable inspection techniques are able to detect flaws to some minimum value. Although a component may not contain flaws as large as the minimum detectable value, it will be conservatively assumed for classification purposes that there does exist at least one flaw at this minimum detectable value, a_m .

Q - The Geometry Factor

In addition to the flaw size, its orientation, type and relation to the ligament length are also important. In all cases it is assumed that the flaw is oriented perpendicular to the stress field, that is, in the opening mode of loading. Four types of flaws are considered:

- (a) Through-edge flaws
- (b) Through-center flaws
- (c) Part-through surface flaws
- (d) Internal flaws

Figure 3 shows these types of flaws and gives the corresponding Q-factors for each. It can be shown (Figure 4) that flaw types (a) and (b) are extensions of types (c) and (d), respectively, any may be used as conservative approximations for part-through and internal flaws. Furthermore, assuming that the minimum detectable flaw, a_m , is independent of the type of flaw (i.e., edge or center flaw) it can be conservatively assumed that a_m is associated with a through-edge flaw (since the characteristic center-flaw size is $2a$.) Making these assumptions, for classification purposes, only an estimate of a_m for one type of flaw need be obtained and the geometry factor may be taken to be 1.1.

For cases where the structure is of such a geometry that the characteristics flaw size approaches the ligament length, a modified Q-factor should be used. Table I lists these values for through-edge flaws.

SUMMARY OF CLASSIFICATION PROCEDURE

The following procedure outlines the method of determining whether or not a material exhibits brittle tendencies due to flaw sensitivity. The structure geometry, material, and environmental conditions are assumed to be known.

1. Determine K_i (or K_{IC} , K_{IH} , K_{IC} , K_{TH} , etc. where applicable). Values should be obtained from reliable experimental data.
2. Estimate the minimum detectable flaw size, a_m .
3. Determine the geometry factor, Q . Use 1.1 unless the characteristic flaw size approaches the ligament length, then use Table 1.
4. Calculate the critical stress intensity, σ_c , from Eq. (1).
5. Compare σ_c with the material yield strength σ_{ys} . If $\sigma_c \leq \sigma_{ys}$ the structure should be analyzed using linear elastic fracture mechanics and approved statistical fracture mechanics data should be obtained. If $\sigma_c > \sigma_{ys}$ the material does not exhibit brittle tendencies due to the presence of undetected flaws and fracture mechanics should not be used.

METHOD OF ANALYSIS FOR BRITTLE MATERIALS

As previously noted it is necessary to insure that the method of analysis as applied to a structure is in agreement with the material classification. Fracture mechanics methods shall be used for flaw sensitive materials. Other methods of analysis, including the Weibull statistical method may be particularly well suited for other materials and configurations. A description of these methods follows:

Fracture Mechanics Method

Having defined a material to be flaw sensitive by the previous classification procedure the structure shall be analyzed as follows:

- (1) Determine the critical stress areas

The method of fracture mechanics is applied to particular areas of the structure where the possibility of unstable crack propagation exists. These areas are determined by reviewing (a) the gross elastic stress distribution throughout the structure and (b) the local environment (including temperature, pressure, irradiation, surrounding fluid atmosphere, etc).

The gross elastic stress distribution is defined as the stress calculated without the presence of a crack from classical analytic models or finite-element techniques.

- (2) Determine the statistical minimum K_i

The initial critical stress intensity factor, K_i , is determined experimentally for the material as a function of operating environment in a statistical manner. K_i values corresponding to the temperature, type of loading and environmental conditions associated with each of the critical areas shall be chosen. The statistical minimum K_i shall be defined as that value which is identified with a 99% probability and a 95% confidence level as described later in this report.

- (3) Determine the flaw types

Having established the critical locations it is necessary for the stress analyst to determine the types of flaws (that is, surface, edge, etc.) that can possibly exist at that location. For design purposes it is assumed that the flaw size is equal to the minimum detectable value.

- (4) Select fracture mechanics model

In order to convert the statistical minimum K_i to an allowable stress value it is necessary to use a fracture mechanics model that approximates, as closely as possible, the stress-geometry situation being investigated. Configurations subjected to linear elastic fracture mechanics analysis will, in general, be converted by the following combinations of stress distribution and geometry:

- a) Uniform stress distribution in semi-infinite and infinite structures
- b) Uniform stress distribution in finite structures
- c) Non-uniform stress distribution in semi-infinite and infinite structures
- d) Non-uniform stress distributions in finite structures

Most fracture mechanics models can be represented mathematically by the expression given in Eq. (1). Geometry variations are accounted for by the term Q . For case (a) above, the Q -factors are of the form given in Figure 3. For case (b), where the crack propagation mechanism is influenced by the structure boundary, Q -factors are of the form given in Table 1. Additional Q -factors are available in reference 3.

Since the number of fracture mechanics models associated with non-uniform stress distributions is rather limited they may be developed by approximate methods. For infinite and semi-infinite structures, case (c), the Q -factor is taken to be 1 and the characteristic stress is approximated as defined in paragraph 6.

- (5) Apply modeling factor

The critical stress intensity, σ_c , shall be calculated from Eq. (1) using the parameters as described above. In order to account for modeling errors a factor of 80% shall be applied to σ_c resulting in the stress intensity limit, $\bar{SIL}$. This factor is intended to account for errors associated with selecting and applying analytical models to actual physical situations.

(6) The characteristic stress

Comparison of the stress intensity limit shall be made with the characteristic stress associated with the fracture mechanics model. The characteristic stress is determined from the gross elastic stress distribution. For uniform stress situations, cases (a) and (b) of paragraph 4, the characteristic stress is defined as the uniform stress. For non-uniform stress situations in infinite and semi-infinite structures, case (c), the characteristic stress is defined as follows:

If the gross elastic stress decreases in the direction of anticipated crack propagation, the characteristic stress shall be the mean gross elastic stress over the anticipated crack length.

If the gross elastic stress increases in the direction of anticipated crack propagation, the characteristic stress shall be the value of the gross elastic stress at the midpoint of the critical section.

For case (d) the characteristic stress will be defined along with the model development.

In all cases the characteristic stress is assumed to be statistically distributed. For design purposes, the limiting characteristic stress is defined as the characteristic stress associated with a 99% probability and a 95% confidence level.

(7) Comparison of stresses

The limiting characteristic stress at the anticipated crack location shall not be greater than the stress intensity limit, SIL , at the same location.

Weibull Method

There may be cases where a material is determined to be free of unnatural pre-dominant flaws for a particular structural application and fracture mechanics should not be used. Although fracture may not be associated with one predominant flaw the effect of the existence of natural flaws dispersed throughout the volume of the material may be significant. Weibull's statistical theory of failure hypothesizes the nature of these natural flaws and their distribution. If a material is shown to possess Weibull characteristics, i.e., having the Weibull strength distribution and the corresponding volume effect, the structure may be analyzed by the Weibull statistical method.

The material constants used in Weibull's theory shall be statistically determined from material tests considering the operating environment. The structural reliability at a 95% confidence level, shall be calculated from standard Weibull techniques⁴ using the limit load stress in the structure. This reliability shall not be less than the assessed reliability goal for that particular component.

Non-Linear Elastic Analysis

No elastic-plastic analyses of brittle materials are permitted since most brittle materials are linearly elastic to failure. For those brittle materials having extremely non-linear stress-strain curves a non-linear elastic analysis should be performed. Limit load strains (99/95% statistical values) shall be calculated and the maximum difference between any two principal strain values shall not exceed the allowable strain.

The allowable strain is defined as 50% of the minimum ultimate uniaxial tensile strain. The minimum ultimate uniaxial tensile strain is the 99/95% statistical value associated with the fracture strain from standard test specimens. The effects of cyclic loading and any material anisotropy shall also be factored into the allowable strain.

MARGIN OF SAFETY AND RELIABILITY

Basic Statistical Design

In its simplest form structural reliability should simply be a question of whether the structure will or will not fail. This survival condition can be expressed mathematically as $\mu_m - \mu_L \geq 0$, where μ_L is the load and μ_m is the strength of the structure. However, load and strength are both probabilistic in nature and cannot be represented by a single value. Assume that a large number of material specimens are tested to failure. A plot of the number of observed failures and the corresponding failure stress may appear as shown in Figure 5. If the ordinate scale is altered in such a manner that the area under the frequency curve becomes one, we obtain a similar curve defined as the probability density curve.

Consider the case where only the material strength is described statistically. Such a case may be represented by a known weight suspended on a rod in tension. If the weight is less than the strength of the rod, there will be no failure. However, the strength of the supporting rod will vary from batch to batch and should be described statistically. If the weight were exactly equal to the average strength of the rod, $\bar{\mu}_m$, the probability of failure would be 0.5.

Because the scatter in the material strength is readily obtainable, the average, or mean strength, is not used as the allowable strength. A design minimum strength, S , is defined as that strength above which a certain percentage of the material strengths will fail. If the material strength can be represented by a normal population distribution and it is required that 99% of the material strengths exceed the design minimum strength, the value of S should be located 2.326 standard deviations (σ_m) below the mean strength. In general:

$$S = \bar{\mu}_m - K_m \sigma_m \quad (2)$$

where K_m represents the number of standard deviations between the mean and design strengths of the material.

The choice of a material "design minimum" alone does not necessarily guarantee a satisfactory structural reliability. Another element, called the modeling factor, $\bar{F}$, is usually introduced to lower the design minimum stress to a value defined as the stress intensity limit or $\overline{SIL}$. The value of this factor will depend on the structural application.

In general, the applied load will also be subjected to variabilities and should be described statistically. These variabilities result in a combined stress-strength distribution as shown in Figure 6. The shaded area in Figure 6 is related to the probability of failure (as will be shown in a later section) and is a function of the degree of overlap of the two distributions.

Similar to the design minimum on the strength distribution curve, there can be defined a design limit loading stress, L , below which a certain percentage of the loading stress values must fall. If $\bar{\mu}_L$ is defined as the mean loading stress, the design limit loading stress is given by:

$$L = \bar{\mu}_L + K_L \sigma_L \quad (3)$$

where σ_L is the standard loading stress deviation and K_L represents the number of standard deviations between the limit loading stress and the mean loading stress. This loading stress, L , may be located somewhat below the previously defined $\overline{SIL}$. The dimensionless difference between the $\overline{SIL}$ and L is defined as the margin of safety, $\overline{MS}$, or

$$\overline{MS} = \frac{\overline{SIL}}{L} - 1 \quad (4)$$

Incorporating the previous definitions and referring to Figure 6 the margin of safety can be defined in terms of the mean stresses as

$$\overline{MS} = \frac{\bar{F} (1 - K_m C_{vn})}{(1 + K_L C_{vL})} \frac{\bar{\mu}_m}{\bar{\mu}_L} - 1 \quad (5)$$

The C_v terms refer to the material or load coefficient of variation, i.e., the ratio of the standard deviation to the mean.

Sample Statistics

The quantity of statistical load and strength data available is usually quite limited. Consequently, the statistical parameters (i.e., the mean and standard deviation) must be derived from limited data which can practically be obtained consistent with present cost and time allotments.

The equations previously derived have been entirely in terms of the population mean, $\bar{\mu}$, and standard deviation, σ . In practice, these quantities are unknown, and it is necessary to estimate their values from the sample mean, $\bar{X}$, the standard deviation, s ,

and the sample size, N . Whereas for a normally distributed population it is possible to say that 99% of the material strengths will lie above a value defined as $\bar{\mu}_m - 2.326 \sigma_m$, clearly the same assertion cannot be made of the value $\bar{X}_m - 2.326 s_m$ based on sample statistics. The proportion of the population included between the limits $\bar{X}_m - 2.326 s_m$ and infinity depends, for example, on whether the sample mean is unusually large, reasonably close to the population mean, or unusually small. The sample standard deviation is also subject to chance fluctuations that affect the proportion included between the limits.

It is clear then that the proportion of the universe included between the limits $\bar{X} \pm Ks$ and $\bar{X}$ infinity is a random variable, and that it is impossible to determine K so that the limits will always contain a specified proportion P of the population. It is possible, however, to determine K in such a way that in a large series of samples from normal populations a certain proportion γ of the intervals will include P or more of the population. γ is called "the confidence coefficient" since it is a measure of the confidence with which we may assert that a given tolerance range includes at least P of the population.

Intuitively, it seems reasonable that the values of K for use with sample means and standard deviations will be somewhat larger than the values used if the true mean and standard deviation are known. Table II presents such factors K for various combinations of confidence coefficient γ , proportion P , and sample size N . For example: if $\bar{\mu}$ and σ are known, the items above $\bar{\mu} - 2.326 \sigma$ include 99% of the population; however, if $\bar{\mu}$ and σ are estimated from a sample of 10 items then $\bar{X} - 3.981s$ is required to include 99% of the total population with 95% confidence.

Oftentimes data are estimated from observations taken from several different lots. If there is significant within lot and among lot variation the estimate of the standard deviation should be based on the degrees of freedom, which will not necessarily be equal to $N-1$, and Table II should not be used.

Basic Reliability

The reliability of a component can be determined from the basic concept that a failure probability exists when a given strength value is exceeded by a loading stress. Since stress and strength distributions are approximated by normal distributions extending to $+$ and $-$ infinity there is always some positive probability of failure. This probability is proportional to the amount of overlap between the tails of the two distributions as shown in Figure 7. Furthermore it can be shown⁵ that the probability of failure is itself a normal distribution and is a function of the means and standard deviations of the two overlapping distributions. This functional relationship is expressed in Figure 8 where

$$X_R = \frac{\bar{\mu}_m - \bar{\mu}_2}{\sqrt{\sigma_m^2 + \sigma_L^2}} \quad (6)$$

The reliability calculated from sample statistics must be associated with a confidence level. The following method based on Satterthwaite's approximation relates the sample reliability and size to a population reliability for a given confidence level. A quantity X_R' is calculated from the sample statistics of the distributions as

$$X_R' = \frac{\bar{X}_m - \bar{X}_L}{\sqrt{s_m^2 + s_L^2}} \quad (7)$$

A value of N' , the equivalent combined number of samples of the joint distribution, is obtained from

$$N' = \frac{(s_m^2 + s_L^2)^2}{\frac{s_m^4}{N_m} + \frac{s_L^4}{N_L}} \quad (8)$$

where N_m and N_L represent the sample sizes used in establishing the material strength and loading stress distribution, respectively. Figure 9 shows the relationship between X_R' , N' , and the population reliability for a 90% confidence level. It should be noted that in all cases the reliability computed from sample statistics is lower than that which would be obtained from the same population statistics. Also, for population statistics, i.e., as $N' = \infty$, Figure 9 gives the same values as Figure 8.

Numerical Example

The following simplified illustration demonstrates how an item or part can be designed to both a specified reliability and to the NERVA program structural design requirements. The material strength distribution obtained from sample tests is characterized as follows:

$$\begin{aligned} \bar{X}_m &= 99,100 \text{ psi} \\ s_m &= 6,710 \text{ psi} \\ N_m &= 15 \end{aligned}$$

The load distribution may be estimated by Monte Carlo techniques or by applying the algebra of normal functions to the analytical model. For this particular example the coefficient of variation of loading stress is taken as 10%. In terms of population statistics we get

$$\begin{aligned} \bar{X}_L &= \bar{\mu}_L \\ s_L &= \sigma_L = .1 \bar{\mu}_L \\ N_L &= \infty \end{aligned}$$

Given a modeling factor of 0.80 and a reliability goal of 0.965, find the mean loading stress that will just meet the design requirements and the reliability goal.

Solution

The minimum design requirements will just be met if the margin of safety is zero. From Eq. (5)

$$\bar{X}_L = \bar{X}_m \frac{(1 - K_m C_{vm}) (\bar{F})}{(1 + K_L C_{vL})} \quad (6)$$

The K values, based on a 99% probability at a 95% confidence level, are obtained from Table II for the sample size indicated, i.e., $K_m = 3.52$ and $K_L = 2.326$. Eq. (6) then gives

$$\bar{X}_L = 99.1 \frac{(1 - 3.52 (\frac{6.71}{99.1})) (.80)}{1 + 2.326 (.10)} = 49.1 \text{ ksi}$$

The reliability calculation requires finding X_R' and N' . From Eq. (7)

$$X_R' = \frac{99.1 - 49.1}{\sqrt{6.71^2 + 4.91^2}} = 6.02$$

Also, from Eq. (8)

$$N' = \frac{\frac{(6.71^2 + 4.91^2)^2}{\frac{(6.71)^4}{15} + \frac{(4.91)^4}{00}}}{15} = 35.3$$

Entering Figure 9, the corresponding reliability is .97 at a 90% confidence level. Since the reliability is greater than the prescribed goal the mean loading stress of 49,100 psi is acceptable. Had the reliability goal not been met, a lower mean loading stress and a margin of safety greater than zero would have been required.

Before applying a reliability analysis it is necessary to know the maximum stresses which so combine at the same point in the component as to result in the failure governing stress. The identification of this critical stress point can usually be obtained by inspection of the most likely points and the stress calculated at each point to determine the maximum. It is important that all six stress components be calculated and the proper stress augmenting factors applied to the components. Calculation of the principal stresses may or may not be required, depending on the form of failure governing criterion used. It is also important to determine the strength including all pertinent degrading factors, which, if exceed, leads to a probability of failure. Finally, the failure criterion most applicable to the particular problem at hand should be selected. The importance of selecting the proper failure criterion is illustrated in the next example. This example considers the

application of design reliability theory as applied to the outer web of the NRX A-6 reflector. The most likely point of failure is chosen to be at the inside surface of the control drum hole at the outer web, as shown in Figure 10. Two theories of failure are considered in determining the structural reliability. One is based on the ultimate tensile strength and the other on the fracture toughness of the material.

The maximum stress and the corresponding stress intensity factor calculated at the outer web of the reflector are 32,000 psi and 9,600 psi $\sqrt{\text{in.}}$ respectively. In order to determine the lower limit on reliability these values are assumed to be line-load limiting values. In the fracture mechanics approach, the outer web is represented by the simplified fracture mechanics model shown along with Table I. The stress intensity factor associated with this particular model is calculated from the expression⁶

$$K_I = \sigma \sqrt{\pi a} f\left(\frac{2a}{h}\right)$$

The irradiated strength properties for the beryllium material are given in Table III. Calculation of the reliabilities for both theories of failure using the methods previously described results in the following reliability numbers for the ultimate tensile strength and fracture toughness approach. The reliability associated with the ultimate tensile strength approach is .9935 whereas for the fracture toughness approach it is .38. This large difference is startling and demonstrates that an inadequate design can occur if the wrong failure criterion is chosen. Beryllium was the NRX A-6 reflector material and this material is classified as brittle for this particular application. The reliability associated with the fracture mechanics approach, which is the appropriate theory of failure for this component, is more conservative and is considered to be the correct structural reliability number for the NRX-A6 reflector. Some cracks occurred in the NRX A-6 reflector confirming this analysis. This example points out the consideration and care that must be taken in selecting the proper theory of failure to be used for a component or part to determine its true design reliability.

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TABLE 1
STRESS-GEOMETRY FACTORS FOR THROUGH-EDGE FLAWS
IN FINITE WIDTH PLATES SUBJECTED TO A UNIFORM STRESS [6]

a/b	Q
0	1.1
0.1	1.15
0.2	1.20
0.3	1.29
0.4	1.37
0.5	1.51
0.6	1.67
0.7	1.88
0.8	2.13
0.9	2.45
1.0	2.84

TABLE 2
FACTORS FOR ONE-SIDED TOLERANCE LIMITS [7]
FOR NORMAL DISTRIBUTIONS

Factors K such that the probability is γ that at least a proportion P of the distribution will be greater than $\bar{X} - Ks$ (or less than $\bar{X} + Ks$), where $\bar{X}$ and s are estimates of the mean and the standard deviation computed from a sample of size N .

$N \backslash P$	$\gamma = 0.90$			$\gamma = 0.95$		
	0.90	0.95	0.99	0.90	0.95	0.99
8	2.219	2.754	3.783	2.582	3.187	4.354
9	2.133	2.650	3.641	2.454	3.031	4.143
10	2.066	2.568	3.532	2.355	2.911	3.981
11	2.011	2.503	3.443	2.275	2.815	3.852
12	1.966	2.448	3.371	2.210	2.736	3.747
13	1.928	2.402	3.309	2.155	2.671	3.659
14	1.895	2.363	3.257	2.109	2.614	3.585
15	1.867	2.329	3.212	2.068	2.566	3.520
16	1.842	2.299	3.172	2.033	2.524	3.464
17	1.819	2.272	3.137	2.002	2.486	3.414
18	1.800	2.249	3.105	1.974	2.453	3.370
19	1.782	2.227	3.077	1.949	2.423	3.331
20	1.765	2.208	3.052	1.926	2.396	3.295
21	1.750	2.190	3.028	1.905	2.371	3.263
22	1.737	2.174	3.007	1.886	2.349	3.233
23	1.724	2.159	2.987	1.869	2.328	3.206
24	1.712	2.145	2.969	1.853	2.309	3.181
25	1.702	2.132	2.952	1.838	2.292	3.158
30	1.657	2.080	2.884	1.777	2.220	3.064
35	1.624	2.041	2.833	1.732	2.167	2.995
40	1.598	2.010	2.793	1.697	2.125	2.941
45	1.577	1.986	2.761	1.669	2.092	2.898
50	1.559	1.965	2.735	1.646	2.065	2.862
60	1.532	1.933	2.694	1.609	2.022	2.807
70	1.511	1.909	2.662	1.581	1.990	2.765
80	1.495	1.890	2.638	1.559	1.964	2.733
90	1.481	1.874	2.618	1.542	1.944	2.706
100	1.470	1.861	2.601	1.527	1.927	2.684
110	1.460	1.850	2.587	1.514	1.912	2.665
120	1.452	1.841	2.574	1.503	1.899	2.649
130	1.445	1.832	2.564	1.494	1.888	2.635
140	1.439	1.825	2.554	1.485	1.879	2.622
150	1.433	1.818	2.546	1.478	1.870	2.611
200	1.411	1.793	2.514	1.450	1.837	2.570
300	1.386	1.765	2.477	1.417	1.800	2.522
500	1.362	1.736	2.442	1.385	1.763	2.475
1000	1.338	1.709	2.407	1.354	1.727	2.430
∞	1.282	1.645	2.326	1.282	1.645	2.326

TABLE 3
FAILURE STRENGTH OF BERYLLIUM

	<u>Ultimate Tensile Strength</u>	<u>Fracture Toughness</u>
Mean Material Strength, $\bar{X}_m$	43,000 psi	9,300 psi - $\sqrt{\text{in}}$
Standard Deviation, s_m	3,930 psi	710 psi - $\sqrt{\text{in}}$
Sample Size, N_m	59	6
Temperature	-230°F	-230°F

Fig. 1. Brittle-Behavior Material Classification.

Fig. 2. Effective Reduction of Critical Stress Intensity Factor Due to Cyclic and Sustained Loading.

Fig. 3. Basic Flaw Types and Corresponding Geometry Factors.

Fig. 4. Relationship Between the Basic Types of Fracture Mechanics Flaws.

Fig. 5. Frequency Distribution Curve.

Fig. 6. Load-Strength Distributions and Relationships.

Fig. 7. Areas for Determining Reliability by the Interference Method.

Fig. 8. Reliability as a Function of Stress-Strength Interference Statistics.

Fig. 9. Relationship Between Sample Reliability and Population Reliability.

Fig. 10. Location of Most Probable Area of Failure.

EFFECTS OF PROOF TESTING ^{and} ANNEALING ON AXF-5Q GRAPHITE

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ABSTRACT

The influence of proof loading at room temperature on the tensile and compressive behavior of AXF-5Q graphite is described. It is shown that specimens which survive a proof test at 90% of the average ultimate strength of the material, have strengths on second loading above the proof level. If the specimens are annealed at 2000°C after proof loading, the stress-strain behavior on second loading is the same as that on first loading. Residual strain effects and dimensional "permanent" sets resulting from proof loading appear to be removed by the annealing process. The results favor the use of proof loading and annealing as a means of raising the lower bound design strength of AXF-5Q structural components.

ACKNOWLEDGEMENTS

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INTRODUCTION

The purpose of this investigation was to experimentally study the effects of proof loading on the subsequent mechanical behavior of graphite. The material studied was AXF-5Q graphite, supplied in two-inch-thick plates by the Poco Graphite Company. The designation 5Q refers to a graphitization temperature of 2500°C. This grade of polycrystalline graphite is fine grained and isotropic with a bulk specific gravity typically between 1.80 and 1.90.

The designer's task in designing components of graphite is made difficult by the fairly large scatter observed in tensile strength measurements.

Figure 1 shows the cumulative frequency of failure measured on 129 tensile specimens of AXF-5Q graphite. We note that the measured strength ranges from 4500 psi to 9500 psi. It is undesirable from the point of view of component efficiency to design to the lower bound when the average strength is in the vicinity of 8000 psi. Proof loading has been proposed (for example in Reference 1) as a means of screening out weak material. The proof test concept is illustrated in Figure 2. The distribution curve for the virgin material, if we assume that the material is unchanged by proof testing, would be shifted to the right (to the curve labeled "proofed material") if the proof test at the indicated level were carried out. For the type of distribution observed in Figure 1, the proof level can be chosen so that only a small percentage of material is rejected while a large gain is made in lower bound strength. For example, if the proof level were selected at 7000 psi, only 13% of the proof tested samples would be rejected.

It should be noted, however, that any factor which changes the character of the strength distribution will affect the economics of proof testing. For example, fracture at a given stress becomes more probable as the volume of the component increases because of the greater chance of finding a critical flaw or weak area within the component. Therefore, when the volume becomes large the fraction of parts destroyed in the proof test at a given stress will also increase.

A potential drawback to proof testing is suggested by Figure 3 which shows a typical tensile stress-strain plot measured upon loading to proof level, unloading, and reloading to failure. There is a permanent set which occurs upon unloading which may in the case of precisely-machined components pose some difficulties. Also, on second loading, the stress-strain response is stiffer than that for the virgin material. This is shown more clearly in Figure 4. An implication is that if proof testing is used as a means of screening material or components, the mechanical behavior of the material will not be the same as that of an unproofed piece unless some means is found for removing residual strain effects. In thermo-structural applications the stiffening of the stress-strain curve after proof load may have a detrimental effect on the mechanical resistance of the material to temperature gradients.

Annealing has been proposed as a means of removing the residual strain and restoring the original stress-strain behavior of the material. The experiments of Seldin (Ref. 2) show that the residual strain is removed and the original

stress-strain response restored after annealing to the graphitization temperature for ten minutes. Annealing to 500°C below the graphitization temperature was shown to remove more than 90% of the residual strain. However, Seldin's results were obtained on ATJ graphite, proof loaded to only about 30% of the ultimate strength. At the outset of the present study, it was not known whether annealing would have similar effects on proof-loaded AXF graphite, particularly after loading to a higher percentage of the ultimate strength.

The study reported here was aimed at answering the following questions with respect to tensile and compressive loadings:

1. How does proof loading, followed by annealing, affect the ultimate strength?
2. Does annealing after proof loading restore the original stress-strain behavior?
3. How do proof loading and annealing affect the dimensions of the specimens?

RESULTS

The approach taken in this study was to proof test specimens from sixteen plates of AXF-5Q graphite to a tensile stress equal to 90 percent of the average ultimate strength. The average ultimate strength of the virgin material was determined by testing to failure four specimens from each of the sixteen plates. After proof test each of the surviving specimens* was annealed at 2000°C (in helium) for one hour, and then was reloaded to failure at room temperature. The specimen configuration is illustrated in Figure 5. Tests were conducted at a crosshead speed of 0.02 inches per minute in a rigidly aligned fixture designed to limit parasitic bending strains to less than two percent of the average strain.

Figure 6 shows the net effect of proof loading and annealing on the stress-strain response of the specimens. There is essentially no change in the strain response, in contrast to the behavior without annealing shown in Figure 4.

Figure 7 shows the effect of proof testing and annealing on the tensile strength distribution. None of the proof tested specimens failed below the proof stress, and the range of strengths obtained is similar to that measured on the virgin material. Figure 8 shows the same data in comparison

* One from each of the sixteen plates

to the "truncated" distribution calculated from the smoothed virgin distribution by assuming all the virgin specimens failing below the proof stress were screened out. The actual distribution of the proof tested specimens is quite similar to the ideal truncated distribution.

Similar experiments were performed with respect to compressive loading. Figure 9 shows a typical stress-strain curve for AXF-5Q obtained upon compressive loading to about 90% of the ultimate strength, unloading, and immediately reloading. Two observations are of interest:

1. With the available accuracy of strain measurement, the shape of the reloading curve cannot really be distinguished from the shape of the original loading curve. Therefore, perhaps annealing is of less importance after compressive proof testing than it is after tensile proof testing. However, the dimensional change after compressive proof testing is not negligible.
2. The compressive unloading curve is slightly S-shaped with an inflection point. This observation is possibly of academic interest here; however, none of the constitutive models discussed at this conference would predict that sort of behavior.

Fifteen compressive specimens, taken from a single plate of AXF-5Q graphite, were proof loaded to 18,400 psi. This represents 90% of the average strength observed on seven specimens from the same plate which were loaded once to failure. None of the specimens failed in the proof test. Upon reloading to failure after annealing to 2000°C, the average strength and strain response appeared unaffected by the proof/anneal procedure. The results in compression indicate that proof testing is probably unnecessary with respect to compressive loadings; however, the lack of detrimental effects upon compressive proof loading and annealing is generally encouraging for the use of proof testing in complex stress-states which might combine tensile and compressive loadings.

In both the tensile and compressive experiments, dimensional measurement of the specimens before and after proof testing and annealing showed that annealing tends to remove completely the residual strain imposed by proof loading.

CONCLUSIONS

The mechanisms of deformation and failure in graphite, and also the mechanisms by which annealing restores the original dimensions and stress-strain response of a once-loaded specimen, are not well understood. The

limited results reported here should be supplemented by further study before the proof test concept is adopted for graphite components. Some factors to be considered are the possibility of damage to the material upon proof loading, the effects of multiaxial proof stress-states, the applicability of mechanical proof loading to components which are thermostructurally loaded, and the ability of proof loading to screen material for components which may fail in fatigue. These considerations are briefly discussed below.

The proof test concept is based on the assumption that the first loading, during the proof test, does not excessively degrade the materials performance on subsequent loading. In an earlier study, Dally and Hjelm (Reference 1) investigated the effects of proof loading specimens of CFW graphite in flexure to about 95% of the median strength level of virgin material. No anneal treatment was used. They concluded that proof loading results in considerable improvement in the strength distribution of the surviving specimens, but that some damage occurs which causes about 4% of the specimens to break slightly below the proof level. However, this basis for inferring damage to the material is suspect because the stress-strain response on reloading after proof may have been different from the original stress-strain response; such a change could affect the relation between calculated stress (based on linear elastic beam theory) and actual stress in a flexural specimen. Nevertheless the existence of some damage due to proof loading may also be inferred from the deterioration of strength in fatigue reported by Dally and Hjelm. In the present study on AXF-5Q graphite, none of the proof tested and annealed specimens failed below the proof level. It is conceivable that annealing removes any damaging effects of prior loading. However, the number of specimens tested is too small to draw the conclusion that no damage occurred.

To be effective a proof test should duplicate the critical stress states expected in the structural application. Proof testing will therefore typically require the imposition of multiaxial stress states because the orientation of pre-existing flaws with respect to the stress field probably influences the initiation of fracture at those flaws. The effects of proof testing multiaxially are not known and, in the absence of mechanistic understanding, should not be assumed to be the same as for uniaxial loading.

Most applications of graphite require its use at elevated temperatures with a significant fraction of the applied stress being the result of thermal strain gradients rather than mechanical loading; the effectiveness of

mechanical proof testing at room temperature as a means of screening materials for such thermostructural applications remains to be established. In thermally stressed components significant stress gradients exist and there may be some practical difficulty in devising proof loading techniques which are capable of applying the correct multiaxial stress-state in each critical region of a component.

In spite of these reservations, the results reported here favor the view that proof testing will prove to be an effective means of permitting the use of higher design stresses in graphite components. Insofar as the strength of graphite is considered to be controlled by pre-existing flaws, the application of appropriately oriented proof-stress states will undoubtedly serve to screen out material which might fail at low load levels. The fact that annealing tends to erase the effects of proof loading on subsequent stress-strain behavior is encouraging in that it allows design calculations to be made using data obtained on virgin material. The results, and also those of Reference 1, imply that any damage induced by proof loading is likely to be minor in comparison to the beneficial effects of screening out grossly weak material.

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Fig. 1. Cumulative Distribution of Tensile Strength, AXF-5Q.

Fig. 2. Proof Test Concept.

Fig. 3. Tensile Strain Response on Reloading, AXF-5Q.

Fig. 4. Effect of Proof-Loading on Tensile Strain Response, AXF-5Q.

NOTES

1. UNSPECIFIED TOLERANCES $\pm .010$
2. UNSPECIFIED SURFACE FINISH 16/

Fig. 5. Tensile Specimen.

AVG & RANGE, 16 SPECIMENS, AXF-5Q PLATE MAT'L

- FIRST LOADING TO PROOF LEVEL
- × SECOND LOADING TO FAILURE AFTER PROOF AND ANNEAL (2000°C)

Fig. 6. Tensile Strain Response of Proofed and Annealed AXF-5Q Graphite.

Fig. 7. Effect of Proof Test on Tensile Failure Distribution, AXF-5Q.

Fig. 8. Strength of Proof-Tested Specimens Compared to Ideal Proof Test Theory.

Fig. 9. Compressive Strain Response on Reloading, AXF-5Q.

Fig. 10. Compressive Load Train.

Fig. 11. Compressive Specimen.

SHORT NOTES

Chairman: *C. R. Kennedy*, Oak Ridge National Laboratory

A TECHNIQUE FOR EVALUATING THERMAL STRESS RESISTANCE

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The purpose of this note is to describe briefly a technique which has been studied extensively and used at Battelle for several years to rate quantitatively the thermal-stress resistance of brittle materials. It is based on establishing a condition of radial steady heat flow through the wall of a hollow cylinder, and measuring the flow that causes the cylinder to fracture.

The heat flow at fracture depends on properties of the material and on the specimen geometry. The geometric or shape factor is determined from specimen dimensions. A material parameter can then be obtained readily as the ratio of the heat flow at failure to the shape factor. It provides a quantitative index for rating materials on the basis of the combined material properties that influence thermal fracture.

The evaluations can be made at different temperatures by changing specimen geometry or by changing the boundaries for heat conditions for heat flow. By this technique, fracture can be initiated over a range of temperatures. The evaluation must be made below the temperature at which creep becomes significant within the time frame of the evaluation.

Figure 1 shows the apparatus used for this evaluation. Typically, the specimen is 1 in. ID, 2-1/2 in. OD, 1/2 to 2 in. in length. The voltage drop in the gage section of the graphite heater element along with the amperage are measured to obtain the heat flow in the specimen. Fracture is observed by viewing the specimen through three windows.

A test is conducted by increasing the power to the heater element step-wise, using small increments, until fracture occurs. As indicated, the heat flow at fracture divided by the appropriate shape factor provides a quantitative index of the thermal stress resistance of the material.

The parameter that describes the resistance of a material and is obtained from the test is given by $R_2 = \frac{\sigma R}{E\alpha}$
where

σ = fracture stress

R = coefficient of thermal conductivity

E = elastic modulus

α = coefficient of thermal expansion.

The underlying theory on which the test is based has been experimentally verified for various ceramics; e.g. alumina, silicon carbide, and diborides. Where good property data are available, calculated values for R_2 agree quite well with experimental values. For graphite, the material contribution determined from the test automatically accounts for any curvature in the stress-strain curve.

Typical values for R_2 in units of calories per centimeter per second are less than 1 for aluminum oxide, around 5 for KT silicon carbide, and about 17 for zirconium diboride. Based on property data, we estimate an R_2 of 70 for AXF-5Q graphite and 150 for ATJ-S. Because of the high heat flow required to fracture graphite, it was necessary to use a notched specimen in testing.

The shape factor for a notched specimen can be determined analytically by using a finite-element computer program. Notched specimens have been successfully used to evaluate diboride materials, for which we needed to reduce the temperature of the test to eliminate creep. The applicability of the technique to graphite has been demonstrated by fracturing a specimen of AXF-5Q well within the range of the available equipment.

The notch we have been using is a 1/8-in.-wide slit parallel to the cylinder axis at the outer surface. Typical slits are between 1/4 and 1/2-in. deep, with a 1/16-in. radius at the root.

There have been many comments at this conference about the lack of property data for use in making analytical predictions of fracture. The situation is even worse in materials development. When trying to develop a material, one usually cannot afford to get all of the property data needed to rate thermal-stress resistance. This technique provides a simple, reliable way to obtain a quantitative rating without prior property information.

It also can be used to experimentally verify analytical predictions. The investigator can predict the conditions for fracture of a specimen, and then conduct an experiment to see whether the prediction holds. Further, the technique is useful for studying the response of materials to thermal stresses, both with regard to their strain response and their fracture mode.

Fig. 1. Thermal Stress Apparatus.

X APPLICATION OF MONTE CARLO TECHNIQUES IN
STRUCTURAL ANALYSIS FOR A NUCLEAR ROCKET

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ABSTRACT

The application of Monte Carlo techniques in the structural analysis of the NERVA nuclear rocket engine reactor is discussed. This unique application of gaming techniques begins with a statistical description (i. e., mean and standard deviation) of the significant input parameters in the nuclear-thermal-structural analysis chain. With the assistance of a random number generator, random values can be selected for a complete set of input parameters and a Monte Carlo case can be calculated. From a series of such cases, statistically defined output parameters are obtained. A "weighted" Monte Carlo approach is followed, currently assuming normal distribution throughout but conveniently adaptable to any cumulative density function that available data might support. The convergence to a stable solution within a relatively small number of random trials is graphically demonstrated for representative component stress calculations.

NOTE: The Nuclear Engine for Rocket Vehicle Applications Program (NERVA) is administered by the Space Nuclear Propulsion Office (SNPO), a joint office of the U. S. Atomic Energy Commission and the National Aeronautics and Space Administration. Aerojet Nuclear Systems Company as prime contractor for the engine system and Westinghouse Electric Corporation as subcontractor for the nuclear subsystem, are developing a nuclear propulsion system for space applications.

The incorporation of probabilistic methods in mechanical component design, as described, for example, by Haugen¹, is a relatively new engineering innovation. The expense of testing the NERVA nuclear rocket engine for reliability has provided a compelling incentive to incorporate such probabilistic theory into the day-to-day activities of the stress analyst. One pace-setting development within the NERVA core analysis not exclusively related to graphite components, but judged to be of significant interest to this audience, is the application of Monte Carlo (random trial) techniques. This unique application of gaming techniques begins with a statistical description (i.e., mean and standard deviation) of the significant input parameters in the nuclear-thermal-structural analysis chain. With the assistance of a random number generator, random values can be selected for a complete set of input parameters and a Monte Carlo case can be calculated. From a series of such cases, statistically defined output parameters are obtained. This process is shown in block diagram form in Figure 1.

A "weighted" Monte Carlo approach is followed, currently assuming normal distribution throughout but conveniently adaptable to any cumulative density function that available data might support. The particular structural analysis model to which the Monte Carlo approach is applied is the so-called RATS program. The RATS model is essentially a gross representation of the NERVA nuclear subsystem which calculates intercomponent loads and displacements and selected component stresses. The primary purpose of the RATS analysis is to provide boundary conditions for the detailed component analysis. The results are also suitable for showing general subsystem trends in system parametric trade studies. The Monte Carlo analytic package consists of a nominal RATS analysis and 14 off-nominal random trials. The choice of the number of trials is a cardinal decision in planning a Monte Carlo analysis. The dependability of the results favors a multitude of random trials (computer cases) while practical considerations, like computer expense, favor a minimum number of runs. The following five figures, selected to represent the best, worst and typical situations, demonstrate the reasonable convergence to a good solution for the probabilistic description of an output parameter as a function of the number of Monte Carlo trials. The presence of an occasional "maverick" should not be entirely disconcerting because such anomalies are always statistically possible. Recall that the calculations are for a 95%, not 100%, confidence level.* The conclusions drawn after about a year of effort with these Monte Carlo calculations is that statistical calculations which are extremely useful to the design and reliability prediction processes are possible with moderate investment of computational resources.

*Some compromise of mathematical rigor in the confidence level calculations is admitted.

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Fig. 1. Schematic of Monte Carlo Structural Analysis.

Fig. 2. Axial Load of Top of Outer Reflector.

Fig. 3. Inner Reflector Q''' Radial Stress at Midband.

Fig. 4. Inner Reflector $E \Delta T$ Axial Stress at Midband.

Fig. 5. Average Fuel Element Elongation.

Fig. 6. Average Fuel Element Axial Displacement.

X STATISTICAL CONSIDERATIONS ON THE STRENGTH OF NUCLEAR
GRAPHITES: CHARACTERIZATION, IRRADIATION, AND DESIGN

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ABSTRACT

Because extreme scatter of fracture stress values is a usual characteristic of graphite, the mean strength is neither a satisfactory basis for design stress considerations, nor an acceptable criterion for property evaluation techniques.

In order to define the strength in terms of a statistical distribution, tensile test measurements have been carried out on randomly selected specimens from a Gilso-carbon graphite. According to the weakest-link concept, the larger the specimen volume, the greater is the probability of finding a very weak link and thus a specimen of low strength. This correlation has been confirmed experimentally and, in the formulation proposed by Weibull has been used for characterizing the mechanical behaviour of graphite.

The same approach has been used to describe the extent and the trend of change of mechanical properties in materials subjected to neutron irradiation. Finally, a very simple attempt has been made, towards the use of the Weibull parameters for the design of graphite components.

INTRODUCTION

The refined analysis techniques needed for design and testing of graphite components for nuclear applications, require significantly more property data than usually available, including fracture and plasticity data, and their change under irradiation. The economical acquisition of sufficient data is handicapped because of the large inhomogeneity of graphite: this scatter of data has forced the designers to select values which do not represent statistically established limits and are largely arbitrary in terms of materials science.

In view of the polycrystalline nature of nuclear graphites, associated with the large variety of structural "flaws" (pores, grains, non-graphitized binder and filler, etc.), the attempt to specify the strength of graphite as a function of failure probability, has been made on the line suggested by Weibull¹. Weibull established a mathematical relationship to account for the mechanical behaviour of a model material containing flaws of random size and distribution. Weibull's methods have been studied and applied by several Authors (2 - 5), both to explore their validity as generalized theory and to apply them to specific stress situations.

The formalism and criteria of these Authors are employed in the paper, "to describe" the experimental results based on uniaxial tensile tests. We are fully aware that, while Weibull's approach represents a satisfactory method for the assessment of "failure probability", it is so far somewhat arbitrary to adopt it as an engineering "failure criterion".

STRENGTH CHARACTERIZATION

In order to characterize the strength of a Gilso-coke graphite in terms of statistical distribution, tensile tests measurements have been carried out on cylindrical specimens randomly drawn from a pre-defined "sample size". According to the weakest link concept, the larger the specimen volume, the greater should be the probability of finding a very weak link and thus a specimen of low strength.

This has been confirmed experimentally and is shown in Fig. 1, where four curves are shown which were obtained from tensile data on four different specimen volumes. The observed strength is plotted versus the corresponding probability of fracture, $S = n/N+1$ (where N is the total number of observations and n is the order number running from 1 to N).

The analysis of these data suggests that the strength decreases with the volume according to a Weibull-type distribution:

$$P = 1 - \exp - KV \left(\frac{X - X_u}{X_0} \right)^m$$

where P is the probability of fracture, K is the loading factor (for uniaxial tension = 1), V is the test volume, and X_u , X_0 and m are parameters describing the distribution function.

The S-shaped curves on the figure indicate the best fit of the equation with the experimental points, for constant values of $X_u = 155$, and $m = 3.4$.

Let us briefly consider the physical meaning of Weibull's parameters. X_u is defined as the stress corresponding to a probability of fracture equal to zero; the interest of characterizing a material by its minimum fracture stress is apparent; a sort of safe limit below which the material "never" goes to fracture. The precision in determining X_u is presently very poor: however the "trend" of variation of this parameter could be very significant (see Irradiation Behaviour).

In the first approximation, m is inversely proportional to the coefficient of variation (ratio in between standard deviation and arithmetical mean) of the strength values. The value of m is then strictly related to the homogeneity of the material: it can be said that increasing values of m hold for increasing quality. From our laboratory experience with several graphite types and grades, the value of m ranges from 2.5 to 12; from this point of view, the quality of the Gilso-graphite, reported on here, could be considered very poor.

X_0 is defined as the stress corresponding to a probability of fracture = 0.63 (for unit volume and $X_u = 0$). This means that, when for a given specimen volume, the three characterizing parameters are given (X_u , X_0 , m), the sum $X_u + X_0 = \bar{X}_{0.63}$ is very close and proportional to the mean (bulk) strength

When testing graphite for strength characterization, attention should be paid to select specimen dimensions which are big in comparison with the microstructure (grain size, pores, etc.) of the material; in Fig. 1 the relatively poor fit of the data relative to the smallest volume, could be accounted for by the effect of Gilso-coke particle size (5-600 μ) in relation to the diameter of the specimens (4 mm).

As a result of this characterization, the specimen volume of 1.4 cm³ (6mm dia x 5-mm) has been selected as standard dimension, when testing the Gilso-graphite for its irradiation behaviour. The material constants for the non-irradiated state are then : $X_u = 155$ Kg/cm², $X_0 = 102$ Kg/cm², $m = 3.4$.

IRRADIATION BEHAVIOUR

In order to test the changes of strength due to neutron irradiation, a number of specimens ranging from 12 to 35 has been irradiated at various temperatures and fluences, in the High Flux Reactor of Petten Establishment of the J.R.C.

Fig. 2 and 3 show how the typical S-shaped curves are modified by neutron irradiation (continuous line represents the non-irradiated material) at high ($\pm 1150^\circ\text{C}$) and low ($\pm 600^\circ\text{C}$) temperatures. At high temperature, the strength first increases then decreases with neutron irradiation: at the highest fluence ($7 \times 10^{20} \text{ n.cm}^{-2}$ Ni-Dido) the bulk strength of this material is far below its original value ($X_u + X_o = 101 \text{ Kg/cm}^2$ compared with 257 Kg/cm^2 for the non-irradiated material).*

At low temperature, this "reversal trend" does not appear and the bulk strength shows a continuous increase.

Furthermore, the value of X_u of the Gilso-graphite decreases rapidly with fluence and irradiation temperature, as is shown in Fig. 4. This indicates a greater risk of fracture at low stresses for the irradiated material; in other words, associated with an increase of the bulk strength, the neutron irradiation, while increasing the strength of the already strong material, is weakening the weaker parts of it.

It is apparent that, in addition to the intrinsic difficulty in determining with reasonable precision the value of X_u , its value as a "safe limit" is lost, when dealing with an in-core structure at high temperature.

The value of "m" does not change substantially upon neutron irradiation. In view of the weight of the "m" parameter on structural reliability calculations (see following chapter) this behaviour gives confidence for the use of data from non-irradiated material, to predict the mechanical performance under neutron irradiation.

*This behaviour at high temperature has not to be generalized for all graphites: in some recent experiments with pitch-coke graphites, marked increases (up to 100%) of the bulk strength have been observed at 1250°C for fluences of the order of $1 \times 10^{21} \text{ n.cm}^{-2}$.

STRUCTURAL DESIGN

The implications of extending the Weibull approach to complex stress conditions, so that it becomes a failure criterion relating strength values, are considered.

Two characteristics of graphite cause conventional methods for determining actual stresses in complex structures to be inadequate: 1) strengths are statistically distributed over a relatively wide range and 2) the range over which they are distributed depends on the size of the specimen and the stress state to which it is subjected.

The most widely accepted reason for the scatter in strength values is obtained from the flaw theory of fracture (4). The theory considers two general cases. One case says that failure at fracture of the single most severe flaw results in total fracture of the specimen: this is the weakest-link theory by analogy to a chain. The other case says that total fracture need not result from a single flaw: failure of a single flaw can result simply in a redistribution of stresses, total fracture occurring when the remaining material in the specimen is no longer capable of carrying the redistributed load. The weakest-link theories are the most widely accepted, and, in the formulation proposed by Weibull, have proved to explain the size effect on Gilso-graphites when tested under uniaxial tension.

The stress-state effect can be considered as a derivation of the size effect. This could be visualized by considering a beam subjected to pure bending: as the nominal tensile stress varies linearly from zero at the neutral axis to a maximum at the outer surface, a severe flaw near the neutral axis would be less effective in causing fracture than would a moderate flaw near the surface.

In accordance with the effect of size, specimens subjected to pure tensile stress do have a lower mean strength and have strengths distributed over a smaller range than do identical specimens subjected to bending.

Freudenthal⁴ has shown that the general weakest-link concept can be treated quantitatively by statistical methods. Suppose it is desired to predict the distribution of strength among a group of specimens cut from a block of material which contains numerous flaws. Because the strength of each specimen will be limited by the most severe flaw it contains, it is necessary to find the distribution of most severe flaws among the specimens: this distribution can be calculated if the distribution of the flaw severities in the parent body is known. Conversely, if the distribution of most-severe flaws (or ultimate strengths) for the specimens is known, it is possible to deduce the distribution of flaw severities in the parent body.

In other words, if the same dependence of the strength level on volume of material can be assumed for tensile specimens and parent body, the reliability of a graphite structure subjected to a definite state of stress could be calculated as a function of the characterizing parameters (m , X_u , X_o) of the material.

In order to put in evidence the specific influence of Weibull parameters, a very simple calculation is presented. Consider a graphite beam 30x30x800 mm loaded with 9 Kg in the middle (giving rise to a maximum bending moment of 40 Kg/cm²) and simultaneously stressed in tension by 65 Kg/cm². The calculation is made assuming two materials A and B characterized by:

$$A) \quad m = 3.4 \quad - \quad X_u = 37 \text{ Kg/cm}^2 \quad X_o = 212 \text{ Kg/cm}^2$$

$$B) \quad m = 6.3 \quad - \quad X_u = 37 \text{ Kg/cm}^2 \quad X_o = 148 \text{ Kg/cm}^2$$

The results of this calculation (for the detailed method see the Appendix) show that, under the assumed load, the beam does have a probability of survival of 78% when the graphite B) is used; if the graphite A) is used, the reliability is reduced by one order of magnitude (8,6%).

It is worth noting that the bulk strength of graphite A) is higher (249 Kg/cm²) compared to the graphite B) (185 Kg/cm²), thus underlining the primary relevance of the parameter m . The value of X_u has been kept low and identical for the two graphites in view of the specific stress level: it is evident that, if a value of $X_u = 155 \text{ Kg/cm}^2$ could be safely guaranteed (as it would be the case of the Gilso-graphite previously characterized, when all limitations are removed as far as the precision is concerned), the reliability of the beam would be 100%, by definition.

Using the same entry data, a parametric study has been performed and the result is shown in Fig. 5. It is apparent that the increase of the bulk strength ($X_u + X_o$) does not improve the reliability to acceptable values, when the value of " m " is too low (just indicating a very inhomogeneous material). It may be useful to remember that the two values of " m " are not unrealistic for nuclear graphite: a value of 3.4 has been found in our experiments with Gilso-graphite, 6.3 has been reported in literature for ATJ graphite (6).

CONCLUSIONS

It has been shown that the strength of a Gilso-coke graphite is size-dependent and, that the Weibull's formulation of the weakest-link theory is an adequate tool for the characterization of the material.

It has also been shown that this approach permits an accurate evaluation of the irradiation behaviour, on the basis of relative small number of experimental measurements.

As far as the design is concerned, the example presented here is nothing but a tentative towards understanding the influence of material parameters on the attainable strength of structural components. It seems more and more evident that a design philosophy peculiar of brittle materials i.e. where material strength is treated as a statistical variable (7) has to be developed.

Some of the assumptions used in this paper to calculate structural reliability may turn out much too primitive if used for more complex shapes and stresses.*

However, naive the approach might be considered, it seems to give already enough evidence of a choice between materials. What answer can we in fact give today to the following problem: given a graphite component subjected to a definite state of stress, do we choose material A), stronger but characterized by a larger variance, or B), weaker but more homogeneous?

ACKNOWLEDGEMENT

Acknowledgements are due to Dr. G. STERNHEIM for the encouraging discussions and to Mr. S. LLOYD for his support in revising the manuscript.

*R.J. Price⁸ has recently shown that the volume-effect does not correlate consistently the failure probabilities in four-points bend test samples and in internally pressurized thick-walled tubes with a superimposed axial tensile stress.

Fig. 1. Probability Curves of Four Different Specimen Volumes of the Unirradiated Material.

Fig. 2. Probability Curves Obtained at $\pm 1150^\circ\text{C}$ Irradiation Temperature.

Fig. 3. Probability Curves Obtained at $\pm 600^\circ\text{C}$ Irradiation Temperature.

Fig. 4. Values of X_u at Different Irradiation Conditions.

Fig. 5. Probability of Survival of a Graphite Beam ($30 \times 30 \times 800$ mm, Loaded by 65 Kg/cm^2 Tension and 40 Kg/cm^2 Bending) as a Function of " m ".

APPENDIX

Reliability Calculation Of The Graphite Beam

According to the method employed by C.E. Spruill⁶:
Structure - A graphite beam, 30x30mm in cross section, 800mm long.

Materials - Two graphites characterized by:

$$A) \quad m = 3.4 - X_u = 37 \text{ Kg/cm}^2 - X_o = 212 \text{ Kg/cm}^2$$

$$B) \quad m = 6.3 - X_u = 37 \text{ Kg/cm}^2 - X_o = 148 \text{ Kg/cm}^2$$

Loads - 9 Kg weight at the center of the beam (giving a maximum bending stress of 40 Kg/cm² at the center of the beam), plus an additional 65 Kg/cm² tensile stress homogeneously distributed over the section.

Equations - The Weibull equation has been used in the form:

$$P = 1 - \exp - KV \left(\frac{X - X_u}{X_o} \right)^m \quad \text{and} \quad R = 1 - P$$

where:

P = probability of failure

K = loading factor (1/m+1)

R = probability of survival = 1 - P.

V, X, X_u, X_o and m, have the same meaning as explained previously.

The loading factors for the two graphites considered are K_A = 0,227 and K_B = 0,137 respectively.

Stresses - Due to symmetry, only one side of the beam is considered. The left hand side is divided into 100 mm segments and it is assumed that the average stress in each element acts over the entire segment.

- (1) Bending moments in the beam
- (2) Stresses in segment 4)
- (3) Composite stress in segment 4):
 - a = height of fictitious beam
 - b = height of fictitious part of the beam
 - a - b = real beam

Segment 4) - Constant Axial stress = 65 Kg/cm²
 Bending stress = 35 Kg/cm²
 Situation of linear stress gradient ($K = 1/m+1$)

I) Graphite A) - $m = 3.4$ - $X_u = 37$ - $X_o = 212$ - $K = 0.227$

For the entire fictitious beam a)

$V = 4.3 \times 3 \times 10 = 129$; $KV = 29,28$; $X = 100$; $P_a = 0.38$; $R_a = 0.62$.

For the fictitious part of beam b)

$V = 1.3 \times 3 \times 10 = 39$; $KV = 8.85$; $X = 30$; $P_b = 0$; $R_b = 1$.

reliability of segment 4) = $R_4 = R_a/R_b = 0.62$ (graphite A).

II) Graphite B) - $m = 6.3$ - $X_u = 37$ - $X_o = 148$ - $K = 0.137$

For the same situation of stress: $P_a = 0.08$; $R_a = 0.92$ and
 $P_b = 0$; $R_b = 1$.

reliability of segment 4) = $R_4 = R_a/R_b = 0.92$ (graphite B).

The other segments are calculated accordingly giving rise to the table:

$P = 1 - \exp - KV \left(\frac{X - X_u}{X_o} \right)^m$				<u>Graphite A</u>			<u>Graphite B</u>		
				$m=3.4-X_u=37$			$m=6.3-X_u=37$		
				$X_o=212-K=0.227$			$X_o=148-K=0.137$		
		V	X	P	R	R_a/R_b	P	R	R_a/R_b
Segment (4)	(a)	129	100	0.38	0.62	0.62	0.08	0.92	0.92
	(b)	39	30	0	1		0	1	
Segment (3)	(a)	162	90	0.27	0.73	0.73	0.03	0.97	0.97
	(b)	72	40	0	1		0	1	
Segment (2)	(a)	240	80	0.21	0.79	0.79	0.01	0.99	0.99
	(b)	150	50	0	1		0	1	
Segment (1)	(a)	630	70	0.23	0.77	0.82	0	1	1
	(b)	540	60	0.06	0.94		0	1	

The probabilities of survival of the beam under the applied load is the product of the probabilities of survival of the individual segments squared (for symmetrical considerations)

$$\text{Graphite A) } R_A = (0.62 \cdot 0.73 \cdot 0.79 \cdot 0.82)^2 = 0.086$$

$$\text{Graphite B) } R_B = (0.92 \cdot 0.97 \cdot 0.99 \cdot 1)^2 = 0.78$$

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FABRICATION OF ISOTROPIC FIBROUS CARBON-CARBON COMPOSITES

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ABSTRACT

A technique for fabricating near-isotropic composites from anisotropic, discontinuous fibers and particulate coal-tar pitch has been developed.

Using a unique high-pressure impregnation-carbonization procedure, solids and hollow shells were densified. These composites demonstrated exceptional thermal shock and ablation resistance.

INTRODUCTION

Applications which encounter rather harsh thermal and stress environments require specific combinations of material properties. Very often, the combinations of property requirements exceed those available from monolithic materials, and composite structures must be designed and fabricated.

Carbon-carbon composites emerged to satisfy requirements for ablation and thermal shock resistance that could not be met by polycrystalline graphites or other refractory materials. While structural efficiency can be obtained from anisotropic, continuous-filament composites, near-isotropic, fine-grain, discontinuous fibrous composites appear to have superior ablation and thermal shock resistance.

The objective of this work was to develop a fabrication technique for forming and densifying isotropic carbon composites containing discontinuous, anisotropic fibers with length-to-diameter ratios greater than 100.

The fabrication described contains two critical, if not unique, procedures, namely: (1) the formation of small fiber-binder agglomerates as the basic unit of construction, and (2) the high-pressure impregnation-carbonization procedure.

FABRICATION

Screened carbon fibers ($2 \mu D \times 250 \mu L$), subsequent to a $2,100^\circ C$ heat treatment, and particulates (-100 mesh) of coal-tar pitch^(a) were the basic raw materials. As shown in Figure 1, the fibers and pitch were blended, warm pressed, and ground to form the pseudoisotropic fiber-binder agglomerates. A model of the ideal agglomerate or basic construction unit is shown in Figure 2. Actual agglomerates, as viewed by scanning electron microscopy, are shown in Figure 3. These agglomerates appear to be loosely bound and nearly isotropic, although it is not essential that each unit be isotropic.

Isotropic composites in the form of solid billets, hollow cylinders, and conical shells were fabricated by warm isostatic pressing and pressure carbonization. Typical thermal expansion and sonic moduli ratios for orthogonal directions were 1.10/1.00. As a further demonstration of the isotropic nature of the material, thick-wall cylinders (3" ID x 1" wall thickness) were fabricated and densified to 1.8 gms/cc without failure due to shrinkage stresses.

By varying the processing schedules, composites were obtained with 15, 30, and 35-volume-percent fibers. The high-fiber-content materials (~ 35 vol %)

(a) Allied Chemical Corporation's Grade CP-277-15V

"Reference to a Company or product name does not imply approval or recommendation of the product by Union Carbide Corporation or the U. S. Atomic Energy Commission to the exclusion of others that may meet specifications."

were formed by pressure carbonization to 750° C under a pressure of 12,000 - 15,000 psi, as shown in Figure 4. The isostatically pressed pitch-fiber composite was sealed in a stainless steel container. The coal-tar pitch was converted to a coke, and near-theoretical coking yields (by weight) were obtained. Heating to 1,000° C and graphitizing to 2,700 - 3,000° C reduced the coking yields to about 95 percent of the theoretical value; ie, about 85 weight percent carbon was obtained from a pitch containing about 92 weight percent carbon. Density of the pressure-carbonized and graphitized materials was about 1.2 gms/cc. Liquid pitch impregnation, as shown schematically in Figure 5, was combined with the high-pressure impregnation-carbonization technique (Figure 4) to density the material to the desired fraction of the theoretical density. The densification rate and accompanying changes in the pore spectrum are shown in Figure 6 for a solid billet. Table 1 gives density and porosity data for several of the isotropic composites and materials prepared by other fabrication procedures.

DISCUSSION

Near-isotropic carbon-carbon composites were produced from discontinuous, anisotropic carbon fibers and particulate pitch by the technique shown in Figure 1. To obtain isotropy from the short fibers, a technique was developed for making small (30 to 60-mil) fiber-binder units (Figure 3) which could be molded and compacted into a composite. Thermal expansions and sonic moduli showed that the orthogonal directions within these composites were nearly identical. Isotropic shrinkages during processing was best demonstrated by the rapid densification of thick-wall cylinders. Prior densification studies with such lay-up procedures as spraying,

flocking, and vacuum molding showed that anisotropic shrinkages cause severe radial cracking for thick-wall cylinders.

Only one blend of materials and essentially a single processing schedule are shown in Figure 1, but the flexibility of the technique allows almost any type fiber, binder, or impregnant to be substituted for property adjustment.

It was not an objective of this discussion to present the material characteristics for the isotropic fibrous composites;⁽¹⁾ but, since improved thermal shock resistance and lower recession rates were primary objectives of the material development, some recent data are of interest. Using mean values of thermal conductivity, thermal expansion, and strain to failure determined at elevated temperatures (4,500° F), thermal stress parameters are compared⁽²⁾ for three materials in Table 2. From these mean values, a significant improvement in the thermal stress resistance is evident. A detailed report on the individual properties is being prepared.⁽²⁾

Ablation resistance for this family of composites has been reported to be superior at the higher stagnation pressures.⁽³⁾ Recent results on samples of this isotropic fibrous material at densities of 1.7 and 1.86 gm/cc were obtained in the five-megawatt arc heater at the Arnold Engineering Development Center. The behavior of the isotropic composites at a density of 1.7 gm/cc was about the same as the behavior of "standard" materials, but far superior ablation resistance was obtained for the higher density composite (1.86 gm/cc; total open porosity ~ 11%). The tests were conducted at stagnation pressures of approximately 90 atmospheres and enthalpies of 2,550 - 2,700 Btu/lb.

It appears that these isotropic, discontinuous, fibrous composites are capable of meeting the design requirements of thermal stress and ablation resistance. The fabrication procedure is relatively flexible and can be adjusted as demands increase, but it is necessary that the fabrication be carefully controlled to prevent gross flaws, material inhomogeneity, and loss of isotropy.

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TABLE 1 - EFFECT OF PITCH PRESSURE IMPREGNATION-CARBONIZATION CYCLES ON POROSITY

Part No.	After Cycle Number															Description of Original Part
	Bulk Density (gms/cc)					Porosity (%)					Real Density (gms/cc)					
	0 ⁽¹⁾	1	2	3	4	0	1	2	3	4	0	1	2	3	4	
21-48	0.20	1.21	1.58	1.76	1.91	78.0	20.9	15.72	14.21	4.65 ⁽²⁾	1.60	1.53	1.87	2.05	2.00	Sprayed & Carbonized
IP-58	0.97	1.39	1.69	1.77	1.82	38.10	9.89	6.17	5.59	1.40 ⁽³⁾	1.56	1.55	1.80	1.87	1.85	Pressure Carbonized without Compaction
IP-61	1.16	1.48	1.69	1.75	1.77	28.49	5.84	4.47	3.31	1.64 ⁽⁴⁾	1.62	1.57	1.77	1.81	1.80	Pressure Carbonized with Compaction
IP-49	1.15	NS	NS	1.77	-	27.46	NS	NS	7.72 ⁽⁵⁾	-	1.59	NS	NS	1.92	-	Pressure Carbonized Billet
PT-63	1.05 ⁽⁶⁾	NS	1.81	-	-	NS	NS	15.07 ⁽⁷⁾	-	-	NS	NS	2.13	-	-	Sprayed Fibrous Cylinder
PT-55	1.05 ⁽⁶⁾	1.55	-	-	-	NS	17.18 ⁽⁸⁾	-	-	-	NS	1.87	-	-	-	Sprayed Fibrous Cylinder
CY-77	0.77 ⁽⁶⁾	NS	1.75	-	-	NS	17.86 ⁽⁹⁾	-	-	-	NS	2.13	-	-	-	Dry-Cast Fibrous Cylinder
FF-9	1.60 ⁽⁶⁾	1.80	1.99	-	-	NS	17.57	5.44 ⁽¹⁰⁾	-	-	NS	2.18	2.11	-	-	Flocked & Wound
ATJS	1.82	NS	1.94	-	-	14.75	NS	8.13 ⁽¹¹⁾	-	-	2.13	NS	2.12	-	-	Billet

(1) Starting Material.

(2) 98.69 percent less than ten-micron diameter.

(3) 94.56 percent less than ten-micron diameter.

(4) 92.45 percent less than ten-micron diameter.

(5) 99.54 percent less than ten-micron diameter.

(6) Geometric density of conventionally densified part.

(7) 98.08 percent less than ten-micron diameter.

(8) 99.02 percent less than ten-micron diameter.

(9) 98.89 percent less than ten-micron diameter.

(10) 89.16 percent less than ten-micron diameter.

(11) 99.21 percent less than ten-micron diameter.

TABLE 2 - THERMAL STRESS PARAMETERS

Material	Density (gms/cc)	Comparison Temperature (°F)	Thermal Stress Parameter (TSP) (1) (Btu/hr-ft) (10 ⁵)
IP ⁽²⁾	1.77	4500	21.7
ATJ-S (with grain)	1.82	4500	8.4
ATJ-S (cross grain)	1.82	4500	5.7
AXF-5Q	1.8	4500	0.92

(1) Thermal-stress parameters determined from values for thermal conductivity, K , expansion, α , and strain to failure, ϵ , at the temperature indicated using the equation $TSP = K \epsilon / \alpha$. (Data supplied by I. Auerbach, Sandia Corporation, Albuquerque, New Mexico.)

(2) IP designates the isotropic carbon-carbon composite material.

Fig. 1. Fabrication Procedure for Isotropic Fibrous Carbon-Carbon Composites.

Fig. 2. Model of an Ideal Fiber-Binder Agglomerate.

Fig. 3. Scanning Electron Micrograph of Fiber-Binder Agglomerates (Top, 100X; Bottom, 300X).

Fig. 4. Schematic of Equipment for High-Pressure Impregnation-Carbonization Technique.

Fig. 5. Schematic of Equipment for Liquid Resin Impregnation.

Fig. 6. Typical Pore Distributions for Isotropic Fibrous Carbon-Carbon Composites at Different Densities.

DISCUSSIONS

MATERIAL CHARACTERIZATION

CORRELATION OF PROPERTIES OF MANUFACTURED GRAPHITES

Philip E. Armstrong

C. D. Pears (Southern Research Institute): Can I assume that you might be inferring that the modulus for the coke particle might be in the vicinity of 8×10^6 psi rather than in the vicinity of 2×10^6 psi? I'm thinking now primarily in terms of cracks or crack propagation. What modulus affects the crack, 8 or 2?

Armstrong: The filler particles in this case were highly graphitic, and this 8×10^6 psi modulus was an average of single crystal data, assuming that the particles would consist of a dense aggregate of crystallites, having x-ray density and behaving as single crystals. Does that answer your question?

Pears: I can talk with you about it later.

MECHANICAL AND THERMAL PROPERTIES OF AIRCO SPEER GRADES
FOR NUCLEAR AND AEROSPACE APPLICATIONS

Leo F. Simbeck

E. T. Onat (Yale University): Could you give reasons for the increase in strength with increase in temperatures in your graphites?

Simbeck: I think that, in general, all graphite grades that we have seen increase in strength with temperature. Nels Olesen, do you have a comment?

N. Olesen (Airco Speer): I don't have a comment on why it has increase in strength with temperature. This is one of the properties which you are looking for with these high-temperature applications, and this is one of the selling points of graphite. As to why it really behaves this way, I don't think anybody has ever found an explanation. Am I right, Colt [Pears]?

C. D. Pears (Southern Research Institute): I have never seen a profound treatise on it!

E. L. Foster (Battelle Memorial Institute): There is some fairly recent British work (I don't have the reference in hand) which, under some fairly carefully controlled experimentation, concluded that the increase in strength that was observed is strictly a function of the absorbed gases. If you heat graphite slowly and carefully to high

temperatures in a vacuum, cool it back to room temperature, and observe the strength, it is the equivalent of what you have at the high temperatures. In other words, you can see as much as 40% increase in room-temperature strength if you have previously out-gassed.

Simbeck: Dr. Onat, do you know of graphites that don't increase in strength with temperature?

E. T. Onat: No. Of course most metals lose strength with increase in temperature, so the increase in strength is a very interesting property. One could attribute it to stress relaxation, say, due to softening of the filler, or something? Inhibition of brittle fracture, perhaps, at high temperatures? I don't know. Perhaps gases. But it could be of interest to discover why this is so.

S. A. Bortz (IIT Research Institute): Do you have any data in which your graphite peaks in strength?

Simbeck: The ultimate strength curves which I showed I think pretty well represent the peak of the strength at 2500°C, the end of the curves you saw.

Foster: It does begin to fall off.

Simbeck: Apparently it does; yes.

M. C. Smith (Los Alamos Scientific Laboratory): This effect of gases on strength, I think, is very important. We've seen in hydrogen, for example, that the strength increase with temperature begins at a much lower temperature than it does in other gases. I think the mass transport effect that the British are postulating is probably very important here. A crack-blunting mechanism, perhaps; or a material removal mechanism at the tips of the crack in other cases. But a very strong effect of atmosphere on this high-temperature behavior.

THE MECHANICAL BEHAVIOR OF ARTIFICIAL GRAPHITES AS PORTRAYED BY UNIAXIAL TESTS

W. L. Greenstreet, J. E. Smith, G. T. Yahr, and R. S. Valachovic

T. Y. Chang (University of Akron): Is there a distinct yield limit which can be identified in the stress-strain plot, such that the material can be considered as elastic; linear elastic, say?

Also, a second question: when the stress-strain curve becomes significantly nonlinear, are there any internal microcracks developed in the specimen which might contribute to the nonlinear behavior?

J. E. Smith: As I understand the first question, I believe that there is no generally accepted method for obtaining a "yield point," for this graphite. If there are other comments to that question, I would appreciate hearing them.

C. D. Pears (Southern Research Institute): I think I have seen a precision elastic limit for ATJ and AXF. I have one slide on that tomorrow when I'm talking on fracture. But in the qualitative sense that 10 microinches represents elastic return, I think that there is an elastic range.

J. E. Smith: As to the second question, regarding internal microcracks, we have assumed the microcracking, especially in compressive testing at room temperature. However, we have not tried to observe these cracks directly. Is there any comment on that question?

M. C. Smith (Los Alamos Scientific Laboratory): Yes; we've seen these microcracks at very low strain levels by sectioning, and they have been detected acoustically at very low stress levels, as you may know.

In your cyclic loading and unloading, did you carry the loading to the point where you could detect the volume change, which would correlate with the microcracking effect? If so, did the volume increase, which normally accompanies this sort of strain, disappear during the high-temperature heat treatment that followed? In restoring the properties of the material, are you restoring volume and structure, too?

J. E. Smith: We did not make a specific attempt to quantitatively make this correlation. To answer your question, I suspect that it would be a significant correlation.

E. J. Seldin (Union Carbide Corporation): Several years ago, I did run some compression tests on short cylinders, and used a micrometer, with which I could detect a length change in the direction of compression which I was able to correlate with the residual strain measured using strain gages. I found, after regraphitizing these samples, that that length change was actually recovered. The length of the cylinder did increase to its original length.

Dimensional change in the transverse direction was much too small to measure with a micrometer, so I had no way of checking that.

S. A. Bortz (IIT Research Institut): Don't your cycling experiments, with the annealing, give us a little better insight into the question that has come up before, as to why graphite increases in strength with increase in temperature? You build up microcracks and so on during testing. When you anneal these, obviously the specimen appears to come back to an originally unstrained condition. Doesn't this more or less add a little bit of weight onto the side of the effect of residual stresses on the decrease in strength, as you come down to room temperature?

J. E. Smith: I think it does. I think it was Mrozowski who in 1950 first postulated the idea of residual stresses, the increased strength with temperature being some function of the prior history of the virgin material. I tend to agree with your comment.

R. A. Meyer (McDonnell Douglas Astronautics Company): Have you measured a lower limit to the annealing temperature? This might give further insight as to what these effects might be. Have you found annealing, say as low as 1800°C versus 3000°C?

Seldin: Perhaps I could answer this question. I found that annealing removes the residual strain introduced by the static testing, and that if you go back to the graphitizing temperature you can completely remove all of the residual strain that was introduced during the tests, and then rerun a stress-strain curve and get one that would be identical to the original stress-strain curve.

If you anneal at some temperature below the graphitizing temperature, there is a partial recovery of the residual strain, and then when you do a test the results are intermediate between what you originally got and what you would get on rerunning the test with no annealing.

I found that you get some annealing at every temperature from about 150° on up.

E. T. Onat (Yale University): Could you tell us something about the speed of straining in your tests? Were they slow tests?

J. E. Smith: The strain rate was, I believe, about 0.01 in./in./min. Can you verify this, Bob [Valachovic]?

Valachovic: Yes.

J. E. Smith: That was the basic strain rate that we used. It was neither slow nor fast!

Onat: Did you repeat these tests, say, ten times at a speed of ten times or a hundred times more, to check on the effect of rate of straining on the hysteresis curves?

J. E. Smith: The material that we used had previously been studied with regard to strain rate behavior, and had been found, within a reasonable range of the strain rates used, to be relatively insensitive to strain rate. Therefore, we did not try to evaluate the effect of strain rate in this program.

W. T. Barry (Aerospace Corporation): The engineering implication here (I hesitated to say it, because I'm sure it's in everybody's mind) is that this whole business about reliability of graphites could be put to rest, because it gives you the opportunity to proof test, and then use the stuff over again with some degree of assurance. Is this true?

J. E. Smith: Because of time limitations, I showed only eight figures. In the proceedings there will be approximately 27 figures, and there were some cases in which the samples did not show the same degree of recovery as others.

However, from what we have observed, we feel that in the case of these simple, uniaxial specimens a trend was observed: when a specimen essentially recovers, and repeats the same data after annealing, that specimen can be used for several cycles, and tends to continue to recover well; on the other hand, some specimens fail to recover at the beginning, and no amount of annealing, or testing, seems to really bring them back to full recovery.

This would indicate that if you were going into this sort of study, you might regraphitize a series of specimens; make two tests on them, with an annealing after each test, and then pick those specimens which did exhibit this recovery behavior as the specimens to observe.

Seldin: Mrozowski did propose a theory about 14 years ago, which explains the high-temperature strength of graphite. People have argued as to whether it's true or not. The explanation is basically that when the material cools down from graphitization temperature, large differences in the thermal expansion coefficients with the grain and against the grain give rise to cracks and frozen-in stresses; that as you heat the material, you are essentially relieving these residual strains, and the material therefore gets stronger.

W. C. Morgan (Battelle Northwest Laboratory): Our nondestructive testing people have been doing quite a bit of preliminary work on acoustic emission, as Mort Smith commented. Acoustic emission shows up crack development at very low stresses. I think it is going to be quite a powerful and useful tool in studying the development of stresses, the effect of annealing, and other things on graphites in general.

J. E. Smith: Prior to this study, some acoustical emission work was done on the AGOT material that was one of the three materials used for this study, and we found that for this particular type of graphite (AGOT), the tensile failure in the specimen occurred at essentially the beginning of audible microcracking, whereas in compressive tests, the audible microcracking became denser and denser as we approached ultimate failure. It was significant that of about ten specimens that we tested and listened to, only one of them showed evidence of microcracking prior to the failure of the specimen in tension. But that's specifically for a single graphite.

MECHANICAL AND THERMAL PROPERTIES OF GLASS-LIKE CARBONS

W. G. Bradshaw

J. Jortner (McDonnell Douglas Astronautics Company): By adding graphite flake, do you develop anisotropy in the material?

Bradshaw: No; no evidence of any anisotropy.

M. C. Smith (Los Alamos Scientific Laboratory): May I ask about the mean particle size and size distribution of your graphite addition?

Bradshaw: We haven't made a precise estimate or particle size count on the distribution.

Y. R. Rashid (General Electric Company): The material presented this morning dealt more with the compressive properties of the graphite materials. There were some examples of the tensile properties. However, from a stress analysis point of view, I have not seen practical problems where the material failure in compression is really significant. Most problems that deal with our practical design analyses are failure in cracking, or failure in the tensile zone, or the combination of tension-compression. Now if one can guarantee that the material in service, in actual structure, would behave uniaxially, and also under compression, then some of this uniaxial compression material that has been presented today is relevant.

However, problems are at least two-dimensional in nature, if not three-dimensional, and therefore require failure of properties in three dimensions--at least two dimensions. I would like to have seen, for example, the shape of the failure surface of the material being presented, so that I could do some stress analysis.

Dr. Rowley's comment as to the analyst being forced to guess at some of the parameters is certainly evident here. If I were to use some of this data in stress analysis, I could only use one value for the compression or two values for the uniaxial compression and tension characteristics, but the rest of it, or the failure surface, is completely missing, and the material being brittle, the failure surface is not symmetric with respect to the stress axes. Therefore, is there any data available which can provide for stress analysis? There's a paper perhaps this afternoon which might discuss this. I would like to see some of the people who presented papers this morning, as to this.

E. J. Seldin (Union Carbide Corporation): Most of the testing is uniaxial testing. We do have a paper this afternoon on biaxial testing, but not very much has been done in that area.

In the last figure in the paper, I notice that the strength falls off rather rapidly with time at temperature, and it didn't seem to me that it was approaching any limit! Does it?

Bradshaw: It did look like it was approaching a limit, but we were just assuming that was going to be the limit.

W. G. Krellner (Stackpole Carbon Company): How do you account for the migration of the graphite in the glassy carbon with the heat treatment?

Bradshaw: We believe the process is one of nucleation in grain growth. As I indicated, there is a closed porosity in there, and we have seen, in glass-like carbons, some evidence of migration of carbon atoms in the pores which then deposit preferentially, and apparently form graphitic material. But this appears to be a volume diffusion kind of thing.

G. A. Pinkas (NASA Lewis Research Center): Is this material commercially available, and if so, what is the cost and delivery schedule?

Bradshaw: This material is not commercially available from us. We have furnished interested people with samples at cost, if their application appears interesting. The delivery time is generally about six weeks. The cost depends upon the specific design; the difficulty of fabricating a specific shape, and so forth. As it is now produced, it is a fairly expensive material. It's a research item, as far as we're concerned.

J. L. Cook (Oak Ridge Y-12 Plant): What happened to your gas permeability when you made graphite additions to your glass-like carbon?

Bradshaw: We haven't measured that, but I think the gas permeability would increase, because in scanning electron-micrographs of this material you can see some evidence of some cracks, and so forth, between the filler particle and the matrix.

DYNAMIC BIAXIAL AND ELEVATED-TEMPERATURE PROPERTIES OF ATJ-S GRAPHITE

S. G. Babcock, S. J. Green, P. A. Hochstein, and J. A. Gum

W. L. Greenstreet (Oak Ridge National Laboratory): You showed a fracture surface in two-dimensional stress space. Did you measure the strains associated with the failures?

Babcock: Yes. The strains, of course, are three dimensional in nature, since there is radial strain. I don't show these here. I have results in some of the reports. Basically what it shows is that the material is incapable of any tensile stresses above about 1 to 1.5% strain.

Greenstreet: Do you have the stress-strain diagrams associated with these points? And will the strain data be included in your paper?

Babcock: Yes; it will. [Editor's note: Figure 7 and the sentence referring to Fig. 7 were subsequently incorporated in the paper in response to this question.]

Y. R. Rashid (General Electric Company): I think the point that Greenstreet has brought up is important. If the material is rheologically dependent, I would say does exhibit creep, especially in the high temperature range, then the failure surface is not static. It either shrinks with time or expands with time. And if you characterize it by only stresses, then you are actually underestimating the fracture mechanism of the material.

However, in order to characterize it properly perhaps the measurement of strain should be considered in measuring the failure surface as well, since creep under relaxation will take place at the same time.

Babcock: I think that point is well taken. There is a problem in a biaxial test in measuring strain. If the radial strain has to be calculated from strain gages mounted on the inside and outside of the tube, is insignificant, or small, then the projection of the two dimensional strain (the radial or the hoop strain and the axial strain, as an example) would be easily represented in a biaxial plot. If the radial strain is large, then of course you need a three dimensional plot for biaxial testing, and that becomes a little hairy!

J. G. Crose (Aerospace Corporation): Would you care to comment on why anisotropy didn't occur in your stress failure envelope, or why you didn't show it? Did the data show it? It looked like the data might have shown a little.

Babcock: I speak of anisotropy in terms of the difference between two directions of loading, such as with the grain and against the grain. This is very small, in this material, compared with, say, metals as an example. Maybe for other graphites it's large. The biggest difference, of course, is between tensile and compressive loadings, and that was the primary difference shown there. There was very little if any directional difference in, say, compressive strength between the with-grain and across-grain directions.

C. D. Pears (Southern Research Institute): Even at 6400°, ATJ still has about 3000 psi ultimate. I've never looked at ATJ-S at those temperatures. These are low loading rates. I would think ATJ-S would be probably stiffer than an ATJ.

Two or three people, at least, have observed that at 5500° and above you can change stress rates from, say, 10, 50 psi/sec up to 500 psi/sec, and drastically change the nature of the stress-strain curve, the stiffness, and the ultimate. And yet you saw none of this; is that correct?

Babcock: No; that's not correct. I said we did see this. We saw a drastic increase in strength. The data plotted from the available literature (the dashed line in Fig. 10) indicated that at approximately 6000° little or no strength was observed.

We did not do any long time testing; the longest time was 10° per second heating rate, which is not long compared with the long term data. In our data we observed strengths in the range of 5000 psi at 6000°, as shown in Fig. 10.

E. T. Onat (Yale University): You mentioned occurrence of pitting at high temperatures. Could you tell us something more about it? Is it something like material burning, or waviness developing on the surface, or what?

Babcock: We did not look into this as much as we would have liked to. I think a lot more needs to be done. Originally the idea was brought to us by Bill Barry at Aerospace; that this phenomenon might take place. What we saw was described here on the photomicrographs; specimens that were taken after cooling. During the test, looking through the pyrometer, we could actually see specks appearing to come off of the surface, and thus this pitting action. I would say it was not a necking phenomenon, because in fact we observed this before loading, while heating.

S. A. Bortz (IIT Research Institute): I notice that your static data does show an increase in strength with temperature up to 2500, but except for one series of points you don't seem to show that increase at high strain rates. Is there any explanation for that, or is it just the spread?

Babcock: Perhaps there is a little bit of mix up there. All of the test data shown on that graph were taken at a relatively high strain rate of 10/sec. The only effect we were looking for was heating rate effect, or time at temperature effect. So the strain rate did not enter into it. What we saw was that up to those higher temperatures, the heating rate was not a factor, at least during these times. The strain rate sensitivity was not examined.

Bortz: The point that I was trying to bring out is that you don't seem to show an increase as you go up in temperature.

Babcock: I don't know the answer. Perhaps we can discuss it.

W. T. Barry (Aerospace Corporation): With regard to this particulate emission from the surface, we first observed this in some specimens that Colt [Pears] did for us a number of years ago. Above 5500°F this becomes very apparent, and the texture of the surface is roughened in a manner which is consistent with the grain size of the graphite. At the time we were doing vaporization studies of graphite, which also indicated that we were getting particulate emission. You can vaporize the graphite and do a Tyndall beam experiment and show that a very significant

amount of material is coming off the surface in a static environment with no mechanical force on the material at all. It looks as though it could be aggravated by application of force.

Babcock: What we saw was without load on the specimen.

MATERIAL-DESIGN CONCEPTS FOR FILAMENT-WOUND GRAPHITE-GRAPHITE HEATSHIELDS

T. R. Guess and C. W. Bert

R. A. Meyer (McDonnell Douglas Astronautics Company): I was most interested in your deposition conditions. Where was the source of the incoming deposition gas?

Guess: In the deposition process of our filament-wound materials we deposited using a temperature gradient method, and the deposition is from the inside surface to the outside surface.

Meyer: Was the source of the gas at the same position, whether you had one atmosphere on the outside and a lower pressure on the inside, or the inverse?

H. M. Stoller (Sandia Laboratories): As Tom [Guess] says, the deposition process is a temperature gradient inside and outside, with the chamber being at a uniform pressure, be it atmospheric or reduced pressure. The concept he is exploring is maintaining the same type of temperature gradient but then, as a function of time for deposition thickness, changing the deposition pressure.

THE MICROSTRUCTURAL EVALUATION OF GRAPHITES FOR THEIR EROSION AND ABLATION CHARACTERISTICS IN AEROSPACE APPLICATIONS

R. A. Meyer, K. M. Kratsch, and R. B. Dirling, Jr.

C. D. Pears (Southern Research Institute): Doesn't that increase in your pore size distribution curve relate more to breaking open the structure and exposing the pores of closed porosity than anything else? I think three or four investigators have shown this around perhaps 1500 to 2000 psi, which is in the level of all force we should expect.

Meyer: Where we see evidence of this increase is, of course, the smaller pore size, so we think it is evidence of an opening of the structure. It may well affect bigger pores, but our effect is for the $< 1\mu$ pore size. So we didn't see a general level change in the total volume.

Pears: Are you opening up the structure at that critical pressure?

Meyer: Yes, we are opening up the structure, as indicated by the upswing at critical pressure.

I. Auerbach (Sandia Laboratories): Once you apply these high pressures and open up these so-called closed pores, have you remeasured the pore volume to see whether there is an overall increase?

Meyer: Yes, we did, and we didn't see a big increase in total volume, which was the point that I was trying to make a minute ago. If we saw a volume increase, it was a slight increase in the large pores and a big increase in the small pores. By large pores, I am talking about pores in the 5 to 10 μ and larger size range.

CHARACTERIZATION OF PRODUCTION-GRADE H-327 GRAPHITE FOR HTGR DESIGN

H. R. W. Cobb, G. B. Engle, and S. J. S. Parry

W. L. Greenstreet (Oak Ridge National Laboratory): On one slide you had strain at failure for irradiated graphite. Do you have strain at failure data versus exposure for these graphites?

If you do, what type of specimen did you use, and how was the strain measured?

Cobb: As I said, we have been fairly comparative in our measurements, but unfortunately, when you are comparing the strain at failure, you really want to break the specimen twice to be very happy about what you are doing, and we just use the standard uniaxial tensile test piece, which is 1/2 inch in diameter and 3 inches long prior to irradiation. We compare this with pieces which are irradiated, some with gage diameters as small as 0.2 inch. We find that the ASTM procedures for mounting the specimen between flexible mounts are quite good enough, provided we use only a straight cylindrical specimen. We haven't done enough to get a fix on the differences in strain you might see before and after irradiation, since I think we would have to do quite a number of both unirradiated and irradiated specimens, with all other characterizations being well understood, before we could say much about that.

One thing is clear, and that is that the strain in the lateral direction is higher; and I guess when we measure the strain at failure, we are really measuring the strain that accompanies a coherency of cracks. So I think that one has to clearly understand the orientation of cracks in the given graphite before perhaps one might say what significance there might be between various measurements of strain. I think the measurements of strain were made only with extensometers.

J. C. Schutzler (McDonnell Douglas Astronautics Company): What is the lowest total integrated neutron flux at which you notice any percentage of property changes in reactor type graphites?

Cobb: I think you begin to see consoling, perhaps, improvements in the strength of graphite at probably down to about 1 to 1.5×10^{21} neutrons/cm², and at energies above 0.18 Mev. But maybe this was because we weren't precise enough to look at lower fluence levels. We rather enjoy the fact that irradiation doesn't harm many graphites, so we are quite happy with this. I would point out this is also temperature-dependent, and at the higher temperatures the value of Young's modulus for unirradiated material is slightly lower than for the irradiated material.

Schutzler: Have you ever investigated the effects of proton irradiation?

Cobb: No, we have to assure everybody that the graphite is capable of doing its job, and that we really understand it. So we are not so concerned about protons as we are about neutrons.

CHARACTERIZATION OF SEVERAL TYPICAL POLYGRAPHITES WITH SOME CONVERGENCE ON SOLID BILLET ATJ-S

C. D. Pears

W. L. Greenstreet (Oak Ridge National Laboratory): In your tests, did you find that the stress-strain curve was rate-independent up to 3500°F? And over what strain rate was this true?

Pears: The stress gradient, say 4500°, Bill. Over the stress rate range that we can run, typically 10 to 500 psi/sec. And over that range rate dependence won't show up until about 4500°F.

Greenstreet: You say that you can change the stress-strain curve about 50% at 5000° by changing strain rate. What strain rate range are you talking about there?

Pears: Over the range 10 to 500 psi/sec.

MICROSTRUCTURAL ANALYSIS TO DETERMINE ELASTIC MATERIAL
PROPERTIES OF A FILAMENT WOUND GRAPHITE COM-
POSITE HAVING ORTHOTROPIC CONSTITUENTS

J. H. Biffle

J. Jortner (McDonnell Douglas Astronautics Company): The stresses must be multiaxial. When you say stress concentration, are you talking about the maximum principal stress?

Biffle: Yes, the maximum principal stress in the $T_1 - T_2$ plane.

Jortner: Do you have any plans to incorporate multiaxial criteria to aid in this work?

Biffle: No attempt was made to relate these stress values to a failure criterion. If you were to look at what I'm modeling, I don't think you would want to, because the assumptions are rather gross at that level, as far as the fiber being round is concerned. Modulus data is really the main criterion I was looking for. This stress concentration will be some kind of an average type of value. Failure criteria are only good at the level you really know something about.

T.-L. Weng (Union Carbide Corporation): You said that you assume the value of the Young's modulus and Poisson's ratio in the transverse direction, E_T . On what basis did you arrive at those values?

Biffle: Another investigator at Sandia has done a lot of experimentation to determine the modulus in the axial direction, and we deduced Poisson's ratio and shear moduli from data such as properties of pyrolytic carbon. Also, data on Poisson's ratio for isotropic graphite were some consideration. So basically we were just drawing on past experience as to how these kinds of materials behaved.

Weng: In electron microscopy observation we often find that fracture occurred in the interface between the fiber and the resin matrix. Does your theory predict this failure?

Biffle: I didn't try to predict failure with these stress values. Just tried to get trends on strength between laminas of different fibers.

HOLOGRAPHIC INTERFEROMETRIC STUDIES OF CARBON COMPOSITE CYLINDERS

O. J. Burchett, C. G. Murphy, and C. W. Matthews

C. D. Pears (Southern Research Institute): Do you have to hold that cylinder perfectly still as you pressurize it?

Burchett: Yes.

Pears: What constitutes perfectly still for this purpose?

Burchett: Within a couple of wavelengths of the light, around 50 microinches. That's the main problem of the analysis technique, holding it still. With more than one view, if it does move, we can tell how much, and in what direction. However, a substantial fixture was holding this cylinder, and probably the main trickery is obtaining fixtures that can maintain sufficient immobility.

E. J. Seldin (Union Carbide Corporation): Could you tell us something about the type of laser that you used? How long does it take to take a picture? And how many views do you need to photograph to examine the whole cylinder?

Burchett: The laser was a Spectra-Physics 125 neon laser. A picture takes probably 30 minutes to set up and then each exposure probably takes about 15, most of which is for development of the plate. We can analyze very easily 60 to 70 degrees accurately. So we need enough views to cover the circumference of the cylinder on that basis.

W. G. Gottenberg (University of Colorado): In the composite materials you are examining, one would expect quite small-scale deformation to occur; in fact, smaller in scale than the distance between fringes. So you would essentially have very fine, wiggly fringes that might be masked by those broad fringes that you see. Would you comment on your ability to resolve small deformations, or small-scale deformations superimposed on a major deformation that you see on a surface?

Burchett: If you wish to go to smaller scale, you use optics which make the object appear larger, so that you can see these small ones. It's called microholography. If you wish to, shall we say, trail a very, very large deformation, you make a reference exposure, then load at a certain amount, make another reference exposure, and go on up in a stair-step type fashion. If you are interested in nonlinearities, that is.

J. Jortner (McDonnell Douglas Astronautics Company): In the anomalous region of strain, where you predicted fracture, did you notice anything physical about the composite which explained both the strain and the fracture at that location?

Burchett: No; we did not. In fact, they were very small anomalies. And of course this is more or less brittle material, as you know. And our approach on the whole situation was that we did not know the size, the shape, or even the cause of the anomaly. The only thing we were interested in was to see whether whatever was there might affect the elastic behavior of the material. And evidently it did, in the instances we have looked at. They were very, very small. In fact we would even pick up the machining errors in the cylinder. In the particular one we looked at, the inside bore was actually not a cylinder, but a frustum of a cone. There was a little variation along it, and we could see that in the deformation along the length of it.

ULTRASONIC CHARACTERIZATION AND COMPUTERIZED
ANALYSIS OF GRAPHITE BILLETS

R. A. Baker, D. E. Bishop, and G. C. Stoker

T. K. McKinley (Aerospace Corporation): What size flaw can you pick up using this technique?

Baker: We have not established this completely. We are just starting the program. We have taken data from Southern Research Institute (from Mr. Pears), and some work that AVCO Corporation has done, and so forth. We think we are finding things smaller than 20-mil diameter. We are pretty sure of this. We ran some of these billets up to where we had something like 11,000 discrete indications, and we think we're getting pretty small, there.

P. E. Armstrong (Los Alamos Scientific Laboratory): What's your high frequency for the pulse?

Baker: We're using the 5 megahertz lithium sulfate focused transducer. It's a medium-focus transducer, made by Automation, with a 4-inch focal length in water.

R. A. Meyer (McDonnell Douglas Astronautics Company): Have you done this for other graphites, like ATJ-S, or PO-3, and others; and if so, what size flaws can you see with those graphites?

Baker: We have done it primarily on ATJ-S, and that's the one we think we're doing better than 20 mils on. We have done a few AXF-5 billets, and I think we might be even doing better than that on those. We can run the sensitivity up on this as high as we want to. It just depends on how much computer time we want to take, and when you are paying for the computer, computer time is cheap.

CONSTITUTIVE EQUATIONSMATHEMATICAL REPRESENTATION OF MECHANICAL BEHAVIOR
OF NONLINEAR INELASTIC MATERIALS

E. T. Onat

Y. R. Rashid (General Electric Company): In the state variable space, when you have the combined problem, creep and also plasticity, you went above the curve of h equals constant, which implies that in the stress-strain relationship the stress state could go beyond what you could get in the laboratory.

Onat: Yes.

Rashid: I can't understand that physically. How would you expect the stress-strain curve to be the upper bound for any stress state?

Onat: No.

Rashid: Whether you involve creep, or elasticity.

Onat: No. May I try to answer your question by looking at the supposed physics of the situation in one case. Take a single crystal which has a very simple square lattice structure and which may have a few dislocations. I believe that if you deformed this structure in shear suddenly, the deformation would be essentially elastic, because dislocations would not have time to travel. So upon the sudden deformation initially you would have a deformed lattice in the shape of a nonrectangular parallelogram. When dislocations are present, however, they may move along the glide planes. If one of them moves, the passage over this dislocation would relieve the elastic stresses and would make your parallelogram more like a square, and this could happen while the outside shape of the material is not changing. After the passage of a number of dislocations you would have "little stairs" kind of behavior. But this wouldn't change the average shape of the material. And I'm quite convinced that this is really indeed what would happen if a fast deformation were followed by keeping the deformation fixed. The dislocations pass, and they relieve the lattice of the stress, but the shape remains fixed. This is what I was describing as happening about this line. Did I convince you?

Rashid: I will discuss it afterwards with you.

G. T. Yahr (Oak Ridge National Laboratory): You showed the cyclic results, where we cycle to successively higher stress levels, and said that there was an effect of the previous cycles on the shape of the other hysteresis loops.

Onat: Yes.

Yahr: That isn't true when you are loading to higher and higher stress levels. In other words, you could have left out some of the lower stress cycles, and you would have obtained the same cyclic hysteresis loops.

Onat: I should say, perhaps, that suppose an earlier cycle changes the shape of the next cycle, the orientation of it. Then the indications would be that the two state variables wouldn't be enough, and you would look for three state variables. This is the sort of thinking that I wanted to get across.

C. E. Pugh (Oak Ridge National Laboratory): Perhaps one of our later talks today will have something about memory, which may have some association here.

W. L. Greenstreet (Oak Ridge National Laboratory): Would you mind going through again what kinds of experiments one would look at to examine what direction to go in time dependent behavior.

Onat: In a very general sense, I would say that a series of experiments would be helpful, and perhaps some of my earlier remarks would explain why it is important, would be this: Divide the time of interest into intervals and then make tests with piece-wise linear variation of strain over these intervals; repeat this kind of test changing the intervals and so on; and consider strain histories which are characterized, themselves, by say four parameters.

By looking at strain histories of this type, which involve four parameters, one would be able to get some indication whether the state space is four state variables, or more than four state variables.

I say this on the strength of my earlier work which I didn't mention here, but perhaps the relationship ought to be obvious because, after all, by doing tests of this type I can cover a four-dimensional space, if you want to talk that way, and if I'm trying to see whether our state space is four-dimensional, at least I must make four-dimensional tests.

So a short answer to your question would be tests which involve unloading, reloading, unloading, reloading, not in a cyclic plane necessarily, but something like that.

AN ELASTIC-PLASTIC CONTINUUM THEORY FOR ARTIFICIAL GRAPHITE

W. L. Greenstreet

J. C. Rowley (Los Alamos Scientific Laboratory): Are all of those fourth ranked tensors really meaningful?

Greenstreet: Yes; it looks formidable in the beginning, but it really is not. One can expand those and it comes out reasonably simple.

Rowley: Is it possible that almost anybody who has a nonlinear computer code could input all that information and get good answers back for graphite?

Greenstreet: Insofar as I have examined the theory, I would expect that he would get good answers back.

E. T. Onat (Yale University): I should make a comment about the fourth-rank tensors. If one takes the view that what's happening inside the material element is represented by some parameters, and if one looks at three-dimensional spaces, one quickly discovers that these parameters must have tensorial character. Also they are averages of what's happening inside. For example, in concrete or in graphite you have pores. When the pores deform, due to the deformation of the material, one may like to give information as to the shape of the pores, and that really would involve a tensorial kind of information. Say I want to give average information about the shape of the pores. Let's assume that to begin with they are spherical and that later they become ellipsoidal. One must give some idea about the ellipsoids and that would involve giving three numbers, in a sense; and then perhaps later on one must also average over the ellipsoids in different directions, and that would give rise to tensors of some order.

So the internal situation results in tensors of some rank because we are representing complicated geometric situations, and naturally the tensors must come about.

F. C. Weiler (Aerotherm Corporation): I noticed the form of the yield function that you put on the board, if I am correct, had about 21 independent constants that must be determined. You used a form α_{IJKL} which, after you made the assumptions of stress and strain symmetry, etc., you finally reduce from 81 constants to 21 constants. How much further did you reduce the number of constants that you had to determine before you actually used this form?

Greenstreet: Graphites are usually transversely isotropic, and hence we have five constants. Just in the elastic theory. So we considered a five-constant theory, for the results I showed for comparison. But the more complex the behavior is, the less symmetry there is in an element, the more constants there are to describe that behavior. You aren't saving by short-cutting on constants, because you aren't representing the physical aspects of the behavior.

Weiler: I presume you assumed that the various degrees of strain hardening in the two principal directions were not incorporated in these constants, and that they were the same. In the individual stress-strain diagrams, say in the with-grain and against-grain directions, you will notice that the rate of strain hardening is different in these two

directions, and consequently you will require more than five constants to adequately represent this.

Greenstreet: Yes, you can require more than five constants. But you can also indicate the differences in the strain hardening, as you call it. You have five constants in the elastic theory and five constants in the plastic portion, so that really there are ten material constants for this simple representation. And it does represent the different responses in the different directions.

STRESS-STRAIN PROPERTIES OF GRADE ATJ GRAPHITE UNDER COMBINED STRESSES

Tu-Lung Weng

J. Jortner (McDonnell Douglas Astronautics Company): Your abstract mentions strength data for graphite and comparison with theories; would you care to say anything about that?

Weng: Originally I intended to include that in the talk, but I have run out of time. I do have the slides if anybody is interested in seeing them. [Editor's note: In the paper in the proceedings, the material referred to here is the subsection entitled "Fracture Strength Under Biaxial Stresses," which includes Figs. 8-10.]

C. E. Pugh (Oak Ridge National Laboratory): There apparently is general interest.

Weng: This is a two-dimensional stress plot, tangential stress and longitudinal stress [Editor's note: See Fig. 8.]. The theory shows that tension-tension and compression-tension stress plottings can be predictive, very satisfactorily, but that this was not true of the compression-compression zone. I would say that the reason for this discrepancy here is more the experimental results, rather than the theory. I found I had quite a bit of difficulty trying to test the cylindrical specimen of this type of material in the compression-compression stress state. They seem to have a buckling failure. And I would say that the only possibility of this type of test is maybe to go to solid specimens.

Jortner: With regard to the size of specimens that you tested, could you indicate what sort of wall thickness and diameter, and whether in your strain analysis you considered thick wall effects, since you were measuring at the exterior?

Weng: The diameter is 1 inch, thickness was 0.050 inch. Length was about 3 inches.

Jortner: Did you use a thick wall analysis?

Weng: No, I used Lamé's equations. I think I indicated that.

F. C. Weiler (Aerotherm Corporation): You mentioned that you are using the deformation plasticity theory, or total strain theory, in your formulation. For the particular experiment of the cylinder this assumption is quite good. You almost get radial loading. There isn't much change in the direction of the strain vector from initiation of yield until the final stress state. In general, however, when we look at a three-dimensional geometric object, we obtain path dependence, which automatically excludes radial loading. Do you have any comments about this?

Weng: As I say in my paper, I limit myself to this particular experiment. I would say that if you have to go to a type of loading which is not constant ratio, a theory such as an incremental theory or one involving time dependence would have to be applied here.

THERMOMECHANICS OF RATE-INDEPENDENT MATERIALS WITH MEMORY

R. D. Snyder

C. E. Pugh (Oak Ridge National Laboratory): You noted that the stresses were not dependent upon temperature gradients, whereas the heat flux, of course, was. Does this violate the principles of equipresence, or any such thing?

Snyder: In the form that we've taken it (in fact, in almost all theories), the second law of thermodynamics discards the presence of the temperature gradients, which leads me to suspect the second law of thermodynamics a little.

POSSIBILITIES FOR APPLICATION OF HIGHER- ORDER CONTINUUM THEORIES

F. Essenburg and W. G. Gottenberg

J. C. Rowley (Los Alamos Scientific Laboratory): I don't want to discourage anybody from a new experimental technique, because I think the graphite business needs new ideas, as I said earlier, and so I want to preface my question very carefully that way. What I would like to ask you is, why do you feel that this kind of theory might give us some insight into what graphite materials are doing specifically, when we haven't even been able to wrestle with the gross inelastic part of their behavior?

Essenburg: We're dealing with composites and with plastic behaviors; and in some of the composites we have an exhibition, certainly on the

basis of the photomicrograph, of a gross isotropy; a very weird material behavior. And a local anisotropy which would be characterized by precisely this model that we started with.

Rowley: That seems very reasonable to me.

C. E. Pugh (Oak Ridge National Laboratory): In your constitutive equations, I noticed that you wrote what I will call your couple stress tensor (I'm not sure what it would be called in this theory) as a second-order tensor. It is defined, as I gathered, as some type of limiting ratio of a moment to, say, an area. Moment, of course, is an axial vector; not an absolute vector. So this should give rise to a qualified tensor; not an absolute tensor, I believe. Should this be reflected in the constitutive equation?

Essenburg: Not when we are referring things to a Cartesian coordinate system. You are talking about a relative tensor, and in a Cartesian coordinate system, relative tensor and absolute tensor are identical. In other words, the determinant of the Jacobian is unity.

Pugh: Well, right. It's a matter of plus or minus 1, and should this be reflected in the integrity basis?

Essenburg: In other words, the minus 1 is merely an internal inversion, which we throw out right away. In other words, when we talk about the transformation from one frame to the other, we require that the Jacobian be greater than zero, not merely that it be different from zero.

Pugh: So your isotropy, then, is limited to the proper and not the full orthogonal group.

Essenburg: Right. What you're talking about is the possibility of turning the material inside out, and usually everyone invokes that restriction somewhere along the line. We just put it in at the beginning. This whole thing was linearized very quickly. Of course, you can carry the zeta in a lot of broad generality, and make that linearizing assumption which I made as the second equation much further down the line, if you like, and get to the same result.

Pugh: Thank you.

T. Y. Chang (University of Akron): I'm not familiar with couple stresses, but just curious. When you do the experiments, you have an inhomogeneous stress state. In other words, you have a boundary value problem to solve. It seems to be very difficult to solve these boundary value problems before you arrive at any results.

Essenburg: Yes; there's a great deal here. The difficulty with couple stresses is that it's a very elusive sort of thing, and the boundary condition for the couple stress is that the couple stress vector is zero on the boundary. So if it's going to occur, it's the microstructure

adjacent to the boundary which will evolve the presence of such a couple stress.

The way to excite this, we believe, is to use an externally applied stress field, a vector field, on the surface of the boundary, which possesses an extremely large directional derivative.

I ran out of time, here. One of the things I was going to point out was that the obvious way to produce this is with a knife-edge loading. You can't ask for a larger discontinuity, or a larger gradient in the components of the stress vector applied to the surface, than with a knife-edge loading. However, this also gives you a very large deformation gradient in the vicinity of the application of such a loading.

The experiments we are proposing here have the unique feature of producing this extremely large gradient in the stress components but keeping the gradient of the deformation field well behaved; very smooth.

R. D. Snyder (West Virginia University): I believe this theory has been applied to granular materials. That's been the motivation for much of this work. I think quite a bit already has been done for various granular materials. The couples arising due to the interface interaction between the granules.

Essenburg: Right. It's been looked over. However, in the usual type of experimentation you don't apply these large gradients on the surface.

A FORMULATION OF THE MECHANICAL BEHAVIOR OF ATJ GRAPHITE AT HIGH TEMPERATURE

T. B. McDonough

J. Jortner (McDonnell Douglas Astronautics Company): A constant crosshead speed, in my experience, does not translate to a constant strain rate. In fact, the strain rate appears to continuously increase during such a test, and that may be responsible in part for the deviation between the experimental curve and what your theory predicts at higher strains.

McDonough: Thank you.

J. C. Rowley (Los Alamos Scientific Laboratory): What message got through to your materials test people as a result of your torture?

McDonough: I know what message was presented to them. I don't know what got through to them. I talked to some of our people about doing other testing, and particularly different rates, and loading and unloading, and they were all very interested. Some of them were

interested in doing the testing, but of course what I'm looking at is extremely high-temperature behavior, and it's not the cheapest thing in the world. I haven't got what I asked for.

J. D. Buch (Aerospace Corporation): We did some high-temperature, very short time (30-second) creep tests on carbon phenolics and materials like that. Did the same sort of thing that you're doing; in other words, go back and calculate the strain-stress curves on the basis of that, and it works out real well. The only problem is our design people won't accept that sort of thing. They want the curves!

McDonough: Well, I guess from a design viewpoint, that's going to be the topic of tomorrow's session; especially thermal stress evaluation. We obviously want conservative design looks; linear elastic models or "conservative" at high temperatures. Everybody intuitively feels that way. But I think from recent discussions that maybe the time is coming when we can start looking at other than linear elastic models and at the availability of finite element computer programs and time step approximations.

That's another problem. You come up with a real nonlinear constitutive behavior, and it's hell to do design studies. It's too expensive, for one thing, in terms of computer time. But I think that it's being done more and more. I know people are doing the elastic-plastic kind of calculations.

By the way, in the paper is a finite difference approximation to this formulation, which is appropriate for finite element solutions, and it all turns out, as you know, you have to do time step solutions. You can no longer look at just one time during a reentry, take a temperature distribution and predict the stresses. You have to follow it down, just like the temperature distribution, and it's expensive, but we're getting there.

C. D. Pears (Southern Research Institute): This kind of data is not uniquely any better for a nonlinear analysis than is a regular stress-strain curve. I guess I missed some point. What is the advantage? As a tester, I know at high temperature I would rather get a stress-strain curve and controlled rates, and so on, than obtain creep data. So what am I missing? I appreciate the constitutive point of view, but I don't see the stress analysis tie-in.

McDonough: I guess my message is not a test technique. It's more of a formulation to represent the stress-strain relations. If we look at the design data which was generated at constant strain rates, people have interpreted that in the past and have done analyses using elastic-plastic analyses. These are rate-independent theories. If there is a rate dependence to the material behavior, and it has an effect on a reentry vehicle nose-tip calculations for different vehicles coming in at different speeds, I'm just suggesting that there is a rate dependence to the stress-strain relation. I'm not selling creep tests as ways of doing it, by any means. I think that my remark about stress analysis was simply

that if we do formulate constitutive behavior beyond the linear elastic case which we are all used to, and which we have all used in design studies, it becomes very difficult to do design calculations, and difficult today in terms of expense, I guess, basically. Because with the finite element techniques for actual shapes, you can include almost anything you want, if you have a formulation and put some work into it. But just data is not a formulation.

C. E. Pugh (Oak Ridge National Laboratory): If one considers the special case of Woolley's equation, which is often used to describe uniaxial stress-strain curves, for the particular case of constant strain rate tests, he comes up with a rate-dependent type expression very similar to that presented here. One difference is that it will have a linear dependence on stress, whereas here, as I took it, there was a nonlinear dependence.

VISCOELASTIC REPRESENTATION OF ANISOTROPIC MECHANICAL BEHAVIOR OF GRAPHITE AT ELEVATED TEMPERATURES

C. E. Pugh and J. P. Callahan

Y. R. Rashid (General Electric Company): Your creep curves are obtained under isothermal conditions.

Callahan: Right.

Rashid: Have you looked into the problem that comes up in analyses where you might have variable temperature fields, to see what kind of specific theory you are going to generate in order to apply your formulation to that condition?

Callahan: We have not at this time.

Pugh: As you probably have noticed, the temperature dependence that resulted in this study was quite complicated. It did not resemble anything like a reduced temperature-time kind of parameter, even though if one studied it carefully a pretty strong feature of that could be seen. One would almost have to have something like this to have an integrable type of representation for variable temperatures. However, methods of analysis usually use discrete or step-wise processes, so the analysis program is going to be continually updating the property values in the model, element by element, or whatever. This lessens, I think, any shortcoming that would result.

E. T. Onat (Yale University): I noticed that you took μ_{12} and μ_{13} as constants. Right? Normally, these wouldn't be constants for a transversely isotropic material. You would have other moduli to worry about. Are there any reasons for taking your μ 's as constants? That is to say, not depending on temperature?

Pugh: I think Colt Pears can probably answer that best. I think he ran some spot checks at Southern Research, in obtaining the experimental data used in our study, on trying to measure induced lateral strain. When conducting a uniaxial creep test as I am sure he will attest to, it's virtually impossible to obtain meaningful measurements of lateral creep strains.

C. D. Pears (Southern Research Institute): We had only about a 30 microinch precision on the instrument, and if you calculate the lateral strains for this size specimen, there just wasn't enough signal. The signals are just too small for us to do much with yet. We expect to be able to do it within the next year. We are developing a new analyzer.

Pugh: I think this is the kind of information one would like to have. I agree it's something that needs looking at when we can get the necessary precision in measurements.

T. Y. Chang (University of Akron): In either Nightingale's book or Reynolds' book, the author collected some data for ratio, and it's so scattered you are better off to assume it's a constant.

Pugh: Was that strain ratio data scattered with respect to temperature or time?

T. Y. Chang: With respect to time; not temperature.

Pears: With reference to the applicability of lateral strain measurements, tomorrow I'm going to show some slides which allege to prove that when these graphites are loaded at temperatures of certainly 5000°F or above, the mechanism is appearing at the grain boundaries, so the creep and the strain are carrying a part of the material and opening up the large porosity. From this point of view I don't know how much continuum we have left; it might be a little bit difficult to relate lateral value without going too far.

W. L. Greenstreet (Oak Ridge National Laboratory): On the basis of the data you did have, wasn't the indication that the strain ratio was essentially constant with stress? Even though there was scatter in the data?

Pugh: Yes. That was looked at in detail. By going through the equations one can see that this particular model predicts this constancy. Namely, if one looks at with-grain and across-grain stress-strain curves, for a given temperature, and examines the ratio of the stresses that correspond to a given strain value from these two curves, this is equal to the ratio of the strain ratios μ_{13} to μ_{31} , the parabolic curve in Fig. 3. All the way up the experimental stress-strain curves for the various temperatures which we examined, the consistency and the constancy of this particular ratio was remarkable. To me, at least, this gives strong impetus to the fact that strain ratios are not stress-dependent. Now time-dependence was what we were alluding to a moment ago as not

having as much information on. But the stress dependence we do think can be ignored or is not present.

A VISCOELASTIC REPRESENTATION OF THE BEHAVIOR OF GRAPHITE
UNDER THE INFLUENCE OF NEUTRON IRRADIATION

S. J. Chang

F. C. Weiler (Aerotherm Corporation): You said that the strain versus neutron dose is related in a sense to thermal strain; I notice that it decreased and then increased. The neutron dose is some kind of rate, isn't it? Not so much accumulated over time?

S. J. Chang: When you increase the amount of dose, the volume of the graphite body first decreased, then increased. This happens because the graphite body has been bombarded by neutrons, which destroyed the lattice structure, or something, inside. So it shrinks and then expands, in a characteristic manner.

C. E. Pugh (Oak Ridge National Laboratory): Is it not true, Dr. Chang, that the dose as you used it here is an accumulated dose; not flux?

S. J. Chang: Accumulated dose; yes. It's the amount of energy you put in.

W. L. Greenstreet (Oak Ridge National Laboratory): You are saying that it enters, as does the thermal strain, into the relationship.

S. J. Chang: It enters into the problem as the thermal strain enters into the elastic problem. And certainly the thermal expansion also has been formulated into the problem. So we can also handle them without difficulty.

C. D. Pears (Southern Research Institute): Could you make any comment about similar effects one might get from protons? Do you have any experience with proton exposures?

S. J. Chang: We have only the data for neutron irradiation.

STRUCTURAL ANALYSIS - NUCLEARVISCOELASTIC RESPONSE OF GRAPHITIC MATERIALS
IN IRRADIATION ENVIRONMENTS

T. Y. Chang and Y. R. Rashid

W. L. Greenstreet (Oak Ridge National Laboratory): What do you conclude from the stress-dose profiles?

T. Y. Chang: That's a difficult question to answer. We concluded, in a way, that the maximum stress is of the order of 600 psi throughout the irradiated designed fuel element life. If the material fails near the usual maximum tensile stress for graphite, of the order of 800 psi in the isotropic plane and about 1500 or 1600 psi along the extrusion direction, it seems to us from this analysis that this particular design is O.K.

Greenstreet: Yes. Then you need to know something about the behavior of the structure as a function of irradiation dose, the change in fracture strength.

T. Y. Chang: That is something we don't know yet.

Rashid: If you can tolerate some failure in the specimen, in the fuel block, for example, but would like to control the crack propagation, then you do need the fracture failure surface of the graphite as a function of dose. But as long as your stress level is below the fracture, then you don't need it. But there are cases in which, if fracture develops at operating stress levels, you would like to know how far it's going to propagate.

J. C. Rowley (Los Alamos Scientific Laboratory): You're beautifully trapped by the usual problem. I often wonder sometimes if we analysts should just refuse to analyze anything until we have received all the material properties data we need, but then they wouldn't get the answers they want. I don't think we have much idea from this whether your reactor is going to hold together or not. But I don't apologize. I have done the the same kind of calculations many, many times myself, after which somebody says, "Well, do you think it's going to fail?" And I say, "Well, see my results." And now I list my five pages of assumptions. Now what do you believe? You don't effectively answer anything. What do you do? Strike? Say, "Materials people, make these measurements or I'm not going to analyze"? We heard a gentleman this morning who ran it upside down and backwards, trying to get something out of them, and it proves a point, I guess, like the opening lecture. We can analyze things, given the right properties, and get pretty reasonable looking answers, one suspects. We might even compare those numbers with a reasonable looking failure criterion, if we had one.

I think the challenge I tried to throw out to the materials people is pretty evident.

Maybe the analyst is enamoured with his equilibrium equations, his continuity derivations, and all the rest of it, to the point of failing to make the other issue clear. Perhaps that's true. But I know from my own experience that if I waited until somebody gave me the right data, I probably would never do a single analysis.

A beautiful job, sir.

T. Y. Chang: Thank you, sir.

G. A. Pinkas (NASA Lewis Research Center): Would you care to comment on the failure theory that Gulf General Atomic used in trying to predict whether the reactor would work or not? While you are considering my question, I would like to mention something we did at Lewis. We cranked through pretty much this same problem that you have there, except that we didn't have the neutron density. We had a cold gas coming through a heater, and we were worried about thermal stresses cracking the drilled-core heat exchanger. Based on our assumptions and theories, and so forth, which are subject to the same criticism as anybody else's, we found that the maximum stresses and the worst thermal strains occurred during the transient portion of the curve rather than at steady state. So the point I am trying to make is, I guess you have to decide whether your application warrants a transient analysis or not.

T. Y. Chang: We don't have really a fancy failure theory, which we would like to have. We have just compared the maximum tensile stress at failure for the graphite, in two different directions, and then we perform the stress analysis and come out to maximum stress and see whether this exceeded that maximum stress value or not. If it's a considerable amount under, then we say, "This is probably O.K." If it's very close, then we have to go back to the drawing board and say, "The designer had better do something." This is, so far, the ad hoc approach we have adopted, because we don't have anything more than that.

Rashid: Just in the interest of being fair to the reactor designers, they do not permit any failure of the fuel elements, and if the analysis predicts failure as a function of the neutron dose, they simply reduce the fuel cycle time to maintain complete integrity of the fuel element. This problem came up, however, in the development of fuel particles, and they have to make a major decision to go from loose fuel particles to bonded fuel particles, just based on the failure of the element.

J. M. Corum (Oak Ridge National Laboratory): Usually, the "in" thing to do when people talk about finite element analysis, I guess, is to ask what the band width is, and how long your program runs. I'm not going to ask you those, but I think in this general area of nonlinear analyses there is a lot of interest in how long do realistic and practical problems like this really take.

T. Y. Chang: This kind of problem takes computer running time of the order of 12 minutes, at most. For this type of problem. Because we have to analyze like 20 to 30 time steps, to be able to get our solution. So the cost is not significant in terms of the result you can get. They may not be accurate, but for engineering purposes, sometimes it's O.K.

ANALYSIS OF SPHERICAL COATINGS ON NUCLEAR FUEL PARTICLES

J. W. Prados and H. C. Roland

J. M. Corum (Oak Ridge National Laboratory): As I understand it, these particles are embedded in some sort of a carburized binder, fuel stick, and this all goes in the fuel hole. What effect do you think the binder has on the strength of the coating?

Prados: That's a difficult question to answer, and it's not one that we have addressed in the work that we have done to date. There have been various concepts of the use of coated particles, some of which have involved loose coated particles without a binder material, and some of which have incorporated the use of a binder matrix material. I personally feel that probably with the type of configuration to be used in the HTGR fuel element, where there is a very dense packing of coated particles, a very high volume fraction of the particles, probably the particle-particle interaction would be more important than the particle-binder interaction. Some people here from GGA may wish to take issue with that.

One of the earlier types of HTGR elements that used the coated particles was the spheres, the AVR-type fuel elements, and in those the coated particle density was relatively low, in the vicinity of 30 volume percent in the fuel matrix. In those elements, any interactions that were taking place would have been between the particles and the binder.

E. T. Onat (Yale University): What sorts of failures were observed in your coated particles?

Prados: You mean the physical characteristics of the failures?

Onat: Yes.

Prados: These will be primarily cracks in the coatings. It's rather interesting that in a number of experiments--and this goes back to the very early days of coated particle irradiation--these cracks have been observed sometimes during irradiation tests; the particles will not fail but the cracks will have been initiated, and will have begun to move out through the coating. During experiments where the fast-neutron flux is small relative to the thermal-neutron flux, the primary cause of failure will be internal pressure in the particle, generated by fission gas released from the fuel kernel. Under these conditions the maximum stress would be predicted to occur at the inner surface of the particle

and indeed it's here where the initiation of cracks is observed. However, there have been irradiation experiments performed in high fast-neutron fluxes where an essentially barren kernel has been used, so that there are effectively no fission gas pressure effects introduced on the inside surface. Under these cases one would expect the maximum stress to develop at the outer surface of the coating, because many of these coatings are anisotropic to one degree or another; although they are far less anisotropic than massive pyrolytic carbon deposits. There is a tendency for expansion in the radial direction and contraction in the tangential direction. This produces a maximum stress at the outer surface, and there have been several irradiation experiments where we have actually seen cracks initiated at the outer surface in fast-neutron irradiation experiments. They haven't gone all the way through. But the type of failure that you see is primarily a cracking of the coating.

A MODEL FOR THERMAL AND RADIATION STRESS AND STRAIN IN
VISCOELASTIC, ORTHOTROPIC CYLINDERS, AND ITS
APPLICATION TO BORATED-GRAPHITE ABSORBERS

D. W. Stevens and O. M. Stansfield

Y. R. Rashid (General Electric Company): In view of Dr. Rowley's criticism of introducing various assumptions because of lack of material data, we have two distinct methods of creep analysis, it seems: the one in the previous paper on this problem, and yours. Did you have a chance to compare the two methods?

Stevens: This is really very similar to the previous creep analysis, I thought.

Rashid: The theory is not quite the same. That involves different assumptions, anisotropy, and so forth.

Stevens: I noticed an anisotropy in the elastic properties, but not in the creep properties. Am I right?

Rashid: Well, the question is, there's a chance of trying to compare two different techniques. I'm asking if this has been done, or are you planning on doing it?

Stevens: No; I don't believe that for the absorbers much additional work is going to be warranted.

STRUCTURAL DESIGN OF NUCLEAR ROCKET FUEL ELEMENTS

P. P. Zemanick (presented by R. Bolton)

G. A. Pinkas (NASA Lewis Research Center): Have you ever measured by accelerometer the dynamic loading? What I am thinking of is that, possibly because of the large energy levels in a rocket exhaust system, there might be high degrees of vibration that could cause breakup of elements, and I wonder if this might be a possible cause of the KIWI failures. Would you care to comment on that?

Bolton: Well, luckily you've asked the right person, because my specialty is not fuel elements, but vibrations. We have measured acceleration environments on the pressure vessel. We have also measured acceleration environments on the core support plate. Can you please show the first figure of the next talk? If you recall, the core support plate sits right above the fuel. We measured acceleration vibrations at several places, and we are performing stress analyses, which I will document in some detail, at least on a gross basis, to assure that we have no vibration problems there. We've never picked up any indication of this. We've never picked up any separate element vibrations, and we don't have any indications of gross vibratory problems in NERVA at this date.

Y. R. Rashid (General Electric Company): What is the random variable in your problem? I missed that. Why is this problem nondeterministic?

Bolton: We have to compute a given reliability, and in order to do that we have to compute not only the nominal value of the stress, but its statistical parameters.

Rashid: What's the random variable? Is it the input or is it the failure stress or what?

Bolton: We can take any one of the particular stress mechanisms. For example the heat generation stress, which we had up here. The modulus, of course, has a statistical variance; the alpha has a statistical variance; the conductivity also.

Then of course we have the input for the reactor. The whole control system is set up on the ability to measure a chamber temperature. But of course that's not always so easy to do. So that implies that there is a statistical variance. The whole analysis for the fuel element is done on the basis of an assumed temperature input to the fuel element. That, of course, is the statistical parameter. All of these parameters are really statistical in nature. The world doesn't seem to deal, at least in NERVA, in deterministic variables.

THE NERVA NUCLEAR SUBSYSTEM FRICTION-
DAMPED LATERAL SUPPORT SYSTEM

R. Bolton

G. A. Pinkas (NASA Lewis Research Center): Considering the sensitivity of the system to the friction coefficient, I wonder whether it would be possible to de-couple this by providing the damping in the spring area, rather than trying to determine the graphite coefficient of friction; introduce materials where the coefficient of friction has already been well established and also nonsensitivity to environment, and put this material inside that spring well, so that you can just forget the graphite and choose your angle so that the graphite design becomes nonsensitive.

Bolton: Well, we have two problems here. One is the fact that the core assembly expands to push the spring almost to solid height at the present time. So if we do anything beyond this, we must add more room.

The other feature here is that if we make it something that's hard, it will crush. If we make it something that's soft and flexible, it has a very low friction capability. And yet we are talking about exciting something at very high g's for Saturn, incidentally, nearest the region of resonance. It's something that leads us inevitably to introduce something with high damping, if we can control it.

We have considered this as a possibility, among many other possibilities of design. But it has these space limitations and these questions of getting high damping out of the system.

G. T. Yahr (Oak Ridge National Laboratory): How did you calculate your stresses. With elastic calculations?

Bolton: Yes. The details are shown in the paper, but it is roughly a simple free-body diagram of a space part. We use a computer program, of course, but the details are in the paper. It's straightforward.

STEEL-TO-GRAPHITE CONNECTIONS ANALYZED BY FINITE ELEMENT
TECHNIQUES FOR USE IN NERVA NOZZLE EXTENSION

W. de la Garza

Y. R. Rashid (General Electric Company): How is the bond at the joint between the graphite and the steel maintained?

de la Garza: Preload on a bolt. They are not bonded; they are clamped together by a bolt.

Rashid: Isn't it possible, then, for the clamp to loosen up and cause discontinuities between the two?

de la Garza: Yes, it is. That's what we've been working on. That's the basis of the problem, with orthotropic material clamped against orthotropic material, bearing against steel that's cryogenic, to assure that you do maintain contact at all points. That's the whole problem. If you try to do this with metal, the metal melts if it is not cooled; if it's cooled, you develop horrendous thermal gradients and the stresses are extremely high. With the use of graphite against metal you have the opportunity to mold something to your loads, rather than just making something work with brute force. But it does require quite an analytic process, mostly trial and error, because of the magnitude of the problem.

H. M. Stoller (Sandia Laboratories): You mentioned at the start of your talk that your material was a tape-wrapped graphite. Could you elaborate a bit on what graphite composite you are looking at now?

de la Garza: It's difficult to answer. There are four materials in process. Three are tape-wrapped; one is a continuous-weave, fiber-enforced graphite. I don't know which one is going to be selected. That's another problem.

Stoller: Is this a resin-impregnated carbonized composite?

de la Garza: Yes; it is.

STRUCTURAL ANALYSIS - SPACEANISOTROPIC ELASTIC-PLASTIC STRESS ANALYSIS
OF AXISYMMETRIC SOLIDS

F. C. Weiler

J. C. Rowley (Los Alamos Scientific Laboratory): Why didn't you put your last conclusion, concerning material property characterization, first?

Weiler: I have a graphite; they're using it in nose tips. I have to make an attempt at predicting what's happening. When I examine the problem, I make an assessment for my particular environment that it's an elastic-plastic material. I look at all the material property data that's given to me, and I realize that this data is lacking some vital information. However, I have to make an assessment of what to do. I have to give my boss a number tomorrow. I can't sit around and procrastinate and tell him, "This is graphite, and everyone knows you can't characterize it." Instead I proceed and try to solve the problem, using assumptions and engineering judgement as best I can. I can't ignore what's happening. I realize that we need better descriptions. Until today, I personally feel that we in the reentry vehicle community can take a well-characterized graphite like ATJ-S, for which there is a sufficient amount of test data available for various temperatures, up to about 5500°F, and reliably predict what's happening to our geometric shapes under different loading conditions. I included comments concerning material characterization more or less for the nuclear engineering community. You have problems with neutron dose irradiation; we don't. We have problems, say, above 6000°F, and you don't. However, I personally feel that we can make fairly accurate predictions. Concerning your failure criterion, we have all kinds of failure criteria we look at. One type is a three-dimensional straining criterion. We look at what happens to the material at various temperatures and the combination of strains which finally produces fracture. I could continue with numerous other illustrations, but I personally believe that we can predict graphite performance fairly well. I am not in accord with your thinking that you gave in your Keynote Address, that we're fooling ourselves!

THERMAL/STRUCTURAL ANALYSIS OF DESIGNS
USING GRAPHITE MATERIALS

R. D. Teter

W. L. Greenstreet (Oak Ridge National Laboratory): I was interested in your thermal ratchetting comments. Did you use the isotropic hardening model in your analysis?

Teter: Yes.

Greenstreet: Have you tried a kinematic hardening model?

Teter: Let me explain a little bit more. We had isotropic material properties. We used the Prandtl-Reuss flow equations for this particular model, with kinematic hardening. It did include kinematic hardening. If you go back to the chart--in fact, if you look at the stress-strain curves, you will find that there was some hardening, a small amount, and so we did shift the yield surface. We have done it both ways. It's very easy to do.

T. Y. Chang (University of Akron): With reference to the kinematic hardening, you are taking into account the temperature factor, is that right?

Teter: Yes.

T. Y. Chang: When you have temperature, then, as I saw in your stress-strain plot, your stress reduced as your plastic strain increased.

Teter: Yes.

T. Y. Chang: Do you encounter any numerical problem? And how small does your solution increment have to be to circumvent a problem? To be able to solve it?

Teter: Of course you always encounter the path dependency effects if you take large increments. However, with this particular program, we have found we have encountered very few numerical type problems. This program will handle quite easily, to illustrate the changing temperature and the accounting for the change in temperature. It will handle the hanging-bar problem, where you put on a constant load, say at room temperature, and then later heat it up. The program will show that, yes, you do increase the deflection due to the changing modulus, and of course when you cool it down, it comes back in the opposite direction. But we haven't encountered numerical problems. Of course the increment size, once again, is certainly path-dependent, so you could never take out the nonlinearities of the loading.

STRUCTURAL ANALYSIS OF CARBON AND GRAPHITE FILAMENT WOUND HEATSHIELDS FOR REENTRY HEATING ENVIRONMENT

J. T. Schamaun

T. K. McKinley (Aerospace Corporation): What do you see in this material versus, say, a tape-wrapped carbon-carbon that would make it advantageous over a more conventional "two-dimensional" tape-wrapped material?

Schamaun: Well, I don't really see much difference between the two, from an analysis standpoint. I think your problems are the same. You are going to have layers that are not compatible, temperature-wise. There may be some effect from a practical standpoint. It may be easier in some cases to have a lay-up; in other cases a cylinder-wound structure.

McKinley: But you do feel the residual stress problem would be applicable to any of the carbon-carbons that we might look at in each instance.

Schamaun: If it's in layers; that's right. I think the problem there results from the fact that in the layered material, if the layers are not going to expand and contract together, you're going to have problems. In filament-wound they won't, and if you lay it up with a tape-wrap where the properties of the tape are different in the longitudinal and transverse directions, you will have the same problems. I think that the same problem will be apparent in that nozzle we looked at the other day.

G. A. Pinkas (NASA Lewis Research Center): Where in the cylinders were specimens taken for determining the properties of the material?

Schamaun: The material properties that we used were obtained from individually wrapped cylinders, and the size of the cylinders is the same as that shown in a talk Monday by O'Neil Burchett. It's a cylindrical specimen, I believe about six inches long and three inches in diameter. And they are tested under internal pressure load and axial tension. We tried to generate some data from full-scale cones, wrapped-cone heat shields, and tried to correlate that somewhat with these small cylindrical tests, and there has been a problem there in that it's hard to hold quality control on the large cones.

A THERMOSTRUCTURAL SENSITIVITY ANALYSIS FOR GRAPHITIC NOSE TIPS

R. J. Michalak, T. B. McDonough, and W. Connell

J. Jortner (McDonnell Douglas Astronautics Company): Would you care to comment on how valid you think your results are in view of the fact that you omitted plasticity and Frank Weiler pointed out that that seems to be of paramount importance?

Michalak: Well, it was omitted as an analysis technique. I'll grant that. But you notice the modulus curve that we did use was a degraded modulus curve. So in an engineering fashion, what we've attempted is to take into account some of the high-temperature plasticity effects by using a degraded modulus. What we've done in the past is an elastic-plastic study, and to try to come up with what would be a reasonable representation of an elastic modulus, to get the same results as you would from doing the elastic-plastic analysis.

J. C. Schutzler (McDonnell Douglas Astronautics Company): I would like to point out that the effect of thermal conductivity variations that you predicted is very much dependent on trajectories. In other words, if you had analyzed a different heating rate history, the predicted effect of variations in thermal conductivity would be different from those shown.

Michalak: That's correct.

Schutzler: It's logical, when you think about it, the higher the heating rate, the less dependency on thermal conductivity should result.

Michalak: That's a very good point. I forgot to mention that in the discussion.

J. J. Lombardo (NASA Lewis Research Center): I would like to ask a question about the use of margin of safety in predicting failure. I don't know whether you defined margin of safety, so I really don't know what you mean, but in most cases the margin of safety had a factor of safety in it, and I wondered if this does, and what did you use. And secondly, certainly not a statistical approach to the problem. Margins of safety have been used but reliability people don't like the word margin of safety. It's a term that has been used for many years but not in predicting failures.

Michalak: The failure criterion that we use is comparison of the maximum strains to uniaxial strain. Effectively, what we've done is use a principal strain criterion. We did not consider biaxial stresses, even though the stress state is biaxial. The reason for that is lack of data.

Similarly, I think we are now getting enough data on this shaped ATJ-S that we can do statistical analyses. At the time this was done, the amount of data was limited. If you have only three or four or five data points, what can you do? But the strain data that was indicated here is lower bound data, and I think you will find that since more data has become available on shaped ATJ-S, that particular curve corresponds pretty well in the critical area under 2 sigma variation. And there was no factor of safety included in the margin.

J. L. Cook (Oak Ridge Y-12 Plant): Would you comment a little bit further on the effects of anisotropy in the thermal expansion, and the results on these analyses? Particularly outside the range of ATJ-S?

McDonough: I think, looking at the results of the study, there was no study on the effects of anisotropy. We showed trends of thermal expansion in two directions, but in each case that we studied we took an upper trend with an upper trend, and a lower trend with a lower trend. So we really didn't look at differing anisotropies.

FRACTURETHE DEFORMATION AND FRACTURE MECHANISMS
IN POLYCRYSTALLINE GRAPHITES

M. C. Smith

W. S. Scheib (U.S. Atomic Energy Commission): We were given a little diagram a couple of days ago of a two-spring and dashpot system, and I think your paper covered the first spring, but not the dashpot and the other spring! Do you want to comment on mechanisms associated with those two analogies?

M. C. Smith: I would rather stick to the first spring, I believe, and say that a good deal of this is recoverable. There are residual stresses in this system when it's unloaded, and you do get a restoration of part, at least, of the strain that's observed. In Seldin's work, and other work that has been reported here, they were able to get complete restoration, which I think is possible if you get the strain at low temperature, so there is no diffusion; no irreversible effects.

I think that I could spend more time than you would like on the first spring, and I just don't know what to do with the other spring and the dashpot.

R. A. Meyer (McDonnell Douglas Astronautics Company): The sequence of pictures with those cracks and generation is really great. I'm most interested in, if you do get restoration, annealing effects after stressing. Could you give us some thoughts as to what might be happening? Are those cracks reversible?

M. C. Smith: Yes. This is the chance I've been waiting for-- Would you give me the very last slide, please? This work is all by Walt Green and Gene Zukas and I think they've done a tremendous job. This is a strain material at room temperature after strain has occurred and these cracks have formed between the dislocations and elsewhere in the structure. A kind of random arrangement of them, as you can see, in a kind of normal graphite. What they did then was return it to 2500°C and hold it for a little while, and if you look sharp you will see that this crack, for example, is quite broad in the initial case. After this high-temperature heat treatment it has shrunk down and it hasn't closed up completely, but it has diminished very greatly in breadth, and so has this one, and so have a lot of the other ones, if you begin to look at the detail of the system. In this case they lost something like 25% of the axial strain by this recovery treatment. In other cases it got somewhat greater than that, but it was never complete in their work, because their deformations were done at quite high temperatures, and I think the important thing there is that during the deformation there is

diffusion, a Nabarro-Herring kind of creep that moves material around so that the process is not quite reversible.

J. J. Ferritto (POCO Graphite, Inc.): You probably realize our POCO graphites are a little bit different from ordinary graphites?

M. C. Smith: I do, indeed.

Ferritto: Did you observe the same phenomena you described here with any of our grades?

M. C. Smith: We've never done quite this sort of thing with your grades, but I bet we could! I think that the behavior would be much like this. There is a great deal more randomness in your type of graphite than in most others, and I would therefore expect a really finer detail in the microcracking and probably a better distribution of it. You also are inclined to make graphite with fewer of these big disparate voids in it; less probability of a concentration of this sort of thing. I think the general behavior would be similar to this, because it is, after all, a genuine graphite that you make.

OBSERVATIONS OF FRACTURE PHENOMENA IN POLYGRAPHITES AND THE RESULTING ARGUMENTS OF FRACTURE MODE AND CRITERIA

C. D. Pears

M. C. Smith (Los Alamos Scientific Laboratory): The talk I was going to make would have been concerned with the details of the fracture path between these disparate voids, and I didn't get to make that talk. It does make a big difference, what the nature of the filler particles is, of course, and the nature of the binder residue that's left behind. I have examples which I could show you of high-temperature tests where the fracture was entirely through the binder residue, detouring all of the filler particles, but that's a result of the nature of the filler particles, primarily. Randomness in the internal structure, so that you didn't have these cleavage planes. You can go to the other extreme and in some of our very highly oriented graphites, made, for example, of natural graphite flakes as the filler, it's entirely a split through the filler material except locally where it has to cross a boundary.

Therefore, the influence of both the type of binder and the type of filler is very strong on the fracture path between the big particles, and you've been looking, I think, except for the POCO materials, at kind of one type of material. I think the fracture path might be different in detail for some of the others. Or have you comments on that?

Pears: I haven't looked at high-anisotropy graphites. No comment there. I would expect, though, that if you kept increasing the strength of the binder, you would reach a point where you would be shoving the

crack over to particle bonds and you would run into a limit. You would have to go back and work on the particle.

J. Jortner (McDonnell Douglas Astronautics Company): You mentioned that in ATJ the aspect ratio of the pores was around 4 to 1, or so. Did you notice any preferred orientation of the pores with respect to with-grain or across-grain direction?

Pears: I see an eyeball orientation, but it's not a strong one, and it's on the list to go back and make some counts. I have not made any counts.

Jortner: At high temperature apparently the fracture mechanism you postulate is separation of particles. Wouldn't you expect that you would reduce the anisotropy of strength as you go up in temperature, if that indeed is the case, so that you wouldn't have to go through the particles?

Pears: I haven't thought my way through with that one, frankly. Tentatively, I would agree that the anisotropy should decrease as you got up there.

Jortner: Does that happen?

H. S. Starrett (Southern Research Institute): I think we've noticed at high temperature, in the order of 5500, and 6000, we really don't see any anisotropy.

C. R. Kennedy (Oak Ridge National Laboratory): I find it rather interesting, with respect to the isotropy-anisotropy arguments that you used. Crystallographically, ATJ-S is anisotropic. Mechanically, it is isotropic. Now this casts some sort of doubts on your 45° angle sort of thing.

Pears: That's absolutely right, and that is a phenomenological observation. It's something we want to attack. We have not attacked that at all. The statistical distribution of the particles, for example.

M. C. Smith: We have looked at the arrangement of these disparate voids in molded graphites, which is what I was showing, and I think mostly what Colt has been talking about. There is not much preferred arrangement of these disparates. But when you get into an extruded graphite, they are very likely to link up end to end, and give you void stringers, which are really disastrous to your properties.

Although it's uncommon, it's quite possible to get a preferred orientation of this defect structure which is different from the preferred orientation of the crystallites in the structure. I think that must be what you are seeing in the ATJ-S. There are two different kinds of preferred orientation, you see. The crystallographic thing which we measure with a Bacon anisotropy factor, for example; and the mechanical or electrical or thermal anisotropies. The ratios are usually much larger for the mechanical and physical measurements than from the x-ray microstructure, but they may indeed be in an opposite direction.

Kennedy: What I was trying to say is simply this: If your cracks were Van der Waal type cracks, in other words, cracking between the layer planes, your fracture strength would be dependent on the crystallographic anisotropy which does exist in ATJ-S.

However, if your cracks are not through the filler particles, then they would be more isotropic as you do find in ATJ-S. This suggests to me, really, that your cracks determining the strength are not really those through the filler particles, but in the binder regions, or at least closely associated with the binder.

Pears: I didn't understand; I thought it was the angle you were curious about.

Kennedy: The angles are certainly going to be dependent on the anisotropy. This is what I was meaning to say.

Pears: I'll have to think my way through that.

G. B. Engle (Gulf General Atomic): Reinforcing what Mort [Smith] and Ray [Kennedy] said: If you look at another mechanism for forming cracks that we see in very highly irradiated graphites, in the case of a graphite that is isotropic on a macroscale, but fairly anisotropic on a microscale, when these things expand under high-temperature irradiation, they do form cracks very readily, and as opposed to that, in the case of something that's microscopically isotropic there is a retardation of the formation of cracks under the same conditions. So we see the same thing in irradiation effects; although the mechanism for crack formation is different, the orientation effects I think still are the same as Ray and, I think, Mort have described.

A SIMPLIFIED APPROACH TO PREDICTING THE ONSET OF FRACTURE IN BRITTLE MATERIALS

R. W. Andrae

V. Lungagnani (Commission of European Communities): In correlating the strength of notched and non-notched specimens, did you find a specific influence of the shape of the notch?

Andrae: Yes; there is certainly an effect of the shape of the notch. I found that for a design which has a very sharp notch, in the tensile specimens the effect on the axial load carrying capacity was less than for the design which has the 45° notch. The effect it might have definitely has to be calculated.

Lungagnani: Then in your formulation, maybe, the geometrical shape of the notch does not enter.

Andrae: It affects the stress distribution, which affects the strain energy, which has to be included in the calculation.

Lungagnani: Oh, yes. Then my question is, in your calculation, which is the stress concentration affected, then, at the tip?

Andrae: The stress distribution; yes.

THE MECHANICAL PROPERTIES OF GRAPHITES AND THEIR RELATION
TO A PHYSICALLY BASED STATISTICAL THEORY OF FRACTURE

J. D. Buch

M. C. Smith (Los Alamos Scientific Laboratory): That's a very nice approach to what we're all trying to do. I wonder, however, about the matter of using grain size as the basis of the statistical approach you were outlining at the end, this coincidence affair. You frequently find that within a given grain of graphite, which is initially a particle, you have a lot of variously oriented domains. In the POCO material, for example, you were using the analogy of the POCO material at a grain size of 0.002 inches, I think; 50 microns. They have an optical domain size, which is the size of the region in which the orientation is indeed parallel that's more like 10 microns. There's a small factor that Colt Pears would happily ignore. He's getting to be a regular physicist about a factor of 10! Are you doing the same thing?

Buch: The grain doesn't have to be assumed to be a perfect crystal in order for its effective diameter to cause weak bonding of some sort. That's what I was saying. Weak bonding could extend essentially the extent of the grain due to a poor bonding of the matrix to the surface of the grain--would be good enough, for example, to do it. The other possibility is that the domain size has a degree of preferential orientation within the grain. That would do it, because it would leave essentially an easy fracture path, or easy debonding path across the matrix. There are other possibilities. If the grains are radially oriented with the C planes running radially from the center, right across the middle is essentially a single C plane, and that would also sort of do it. So I was sort of assuming that through any one of those coincidences we should expect the entire grain to be treated as its potential flaw. Not that it was a perfect domain, but for many reasons.

BIAXIAL MECHANICAL PROPERTIES OF AXF-5Q
GRAPHITE TO 4000°F

J. Jortner

J. R. Bohn (TRW Systems, Inc.): Were you able to observe the point at which fracture initiated? Did you try to take movies, or anything of this sort?

Jortner: No; we did not take movies. We relied on reconstructing the specimens after fracture.

T.-L. Weng (Union Carbide Corporation): The strain data I saw are quite similar to what I got. I never reported the data because I was not sure how to interpret the results, because of the apparent odd looking shape. I'm glad to see that somebody obtained the same type of data that I got!

Are you going to test the other stress quadrants?

Jortner: We do not intend to test in other stress quadrants in the near future.

Bohn: Have you tried to plot deformations for other values of strain, to see what a quasi or a pseudo flow surface for the material might look like?

Jortner: Well, I have difficulty in defining yield or the initiation of "flow," because of the nonlinear nature of the stress-strain curves.

Bohn: Yes; I can imagine; but have you tried just to pick arbitrary values of strain and plot that versus stress?

Jortner: We have made some stress-strain plots but have not cross-plotted them to give biaxial flow surfaces in the sense that you may mean.

Bohn: Also, I notice the scatter seemed to be less in fracture at high temperature than at low temperature.

Jortner: At elevated temperature there are less data points. The scatter always seems to increase the more you test!

Bohn: It just seemed like a trend. Do you have any feeling on the subject?

Jortner: There may be such a trend, although I don't think we have enough data to say. Perhaps, because of changes in stress-strain response, one might expect whatever flaws there are to be less effective at high temperatures. Although I think he's talking about higher temperatures than 3000 and 4000°F, Pears' theory that at high temperatures the fracture proceeds between grains, rather than through them, would,

I think, indicate less flaw dependence at high temperature. [Editor's note: Reference 9 was subsequently added to the paper to cite Pears' theory (presented earlier in this same session).] However, possibly the most pertinent factor in the present study is that we did proof test most of the elevated-temperature samples in an attempt to eliminate the weaker specimens.

W. T. Barry (Aerospace Corporation): One thing that struck me as rather peculiar about the scatter in your data was that in almost every case the scatter is almost linear. The data points are not clustered, you know. It's as if you get one data point, and it just keeps moving away from you.

Jortner: Our tests were at definite stress states, with radial loading, and that accounts for the scatter being radial, which makes it appear linear.

C. D. Pears (Southern Research Institute): I think POCO does start getting a little plastic at 4000°F but not at 3000°F. As a matter of fact, at 3000 it's a little stiffer, and that might be punishing it a little bit.

Jortner: Yes; it is stiffer initially, but at higher stresses the tangent modulus at 3000°F seems to be lower (in tension) than at room temperature. I'm not sure what the implications really are, but it seems possible that the difference in tangent modulus near failure might have the predominant effect on the strain field, or the stress field, around flaws. However, any discussion about the effect of temperature on biaxial strength should be regarded as speculative because of the limited amount of data obtained at 3000°F.

T. B. McDonough (General Electric Company): Did you try anything besides proportional loading?

Jortner: No; we did not.

GEOMETRICAL PARAMETERS AFFECTING THE FRACTURE TOUGHNESS OF GRAPHITE

G. T. Yahr and R. S. Valachovic

W. Chard (Pfizer, Inc.): I noticed in your last calculation that the size of the flaw appeared to be approximately the size of the filler particles in the ATJ material. Also, in the POCO material it appeared that that microstructure indicated there were areas of high density, and that size that you calculated appeared to be the size of the high density regions. Do you think there's any relationship between your calculations and those of the filler particles in the high density regions?

Yahr: It's quite possible. I don't know what actually initiates the cracks, whether it's the porosity or the things you mention. Still they are of the order of magnitude to indicate that fracture mechanics will extend down to these small flaws which are actually causing fracture in a structure.

C. D. Pears (Southern Research Institute): So far as I know, nobody has ever been able to relate the smallpox in POCO to the fracture, in any way!

Were you implying by your remark about the natural crack, that you thought that the better the test, the lower K will be?

Yahr: For plane strain fracture toughness, it is lower than for a non-plane strain condition. You can get effects other than arriving at plane strain. For instance, if I had my notch deep enough, I actually got lower values, but this was not an effect of not being plane strain. This was some other effect. For instance, it may have been that the notch was actually deeper than we thought by the natural flaw lengths in the material, and this may have been having an effect at the small notch depths, whereas it didn't at the large notch depths, which would also account for there being more of an effect in ATJ graphite of the notch depth than in the AXM graphite.

J. J. Ferritto (POCO Graphite, Inc.): I think, to clarify the situation a little bit, what you call these discrete flaws in the AXM graphite are not in reality discrete flaws as such in the same sense as they are in the ATJ, but more really they are density inhomogeneities, rather than discrete flaws. So they really can't be compared on the same basis.

Yahr: They seem to correlate with the fracture mechanics concept. I don't know actually whether they're flaws or what they are.

APPLICATION OF WEIBULL STATISTICAL THEORY TO THE STRENGTH OF REACTOR GRAPHITE

R. J. Price and H. R. W. Cobb

J. Jortner (McDonnell Douglas Astronautics Company): Am I correct in assuming that the Weibull curves that you are fitting to data are meant to represent the median strength?

Price: I normalize them all to median strength, just because it's more convenient mathematically, but there's no difference when one uses the mean strength. The fit is essentially the same.

Jortner: However, the design problem would seem to be one of knowing what the lower bound, or very low risk strength is. That is if 50% of the components fail below what you show in the analysis, the analysis

is not very useful. Can you, in fact, on the basis of limited data, as you've done, extend it to the lower bound problem?

Price: Well, it depends what you mean by lower bound. If σ_u is indeed equal to zero, then there is no lower bound. At very low stresses, there will be a very, very small but still finite probability of failure. I guess it depends really on your design philosophy, whether you believe in the existence of a lower bound, or whether you will just settle for a, say, 0.1%, or 1%, or 0.0001% probability of failure.

T.-L. Weng (Union Carbide Corporation): Can you explain to me the accuracy of using the M value from uniaxial tension to predict the biaxial stress condition of the material which is highly anisotropic, and which in this case probably has a different M value in a different direction?

Price: In the case limit area we tested, the M value was not very different in the two directions. In the paper that will be in the conference proceedings, we tabulated these values and they were rather similar for the two directions of testing, although the mean strengths were different. I think under those conditions, it's reasonable to use this calculation for estimating the probabilities of failure under biaxial tension. The one difference would be, however, in the calculated envelope I showed, I assumed that the fracture stresses were isotropic. In an anisotropic case, that envelope would look a bit different. It would be bounded by an oblong rather than by a square. But I think that if the n values characterizing the material are the same for the two directions one can still use the approach.

C. D. Pears (Southern Research Institute): I have a comment that might relieve you! We asked Professor Weibull once if he thought any of these terms were material constants and he hasn't answered us yet. He sat there all day and talked about it, but he never told us that.

THERMAL SHOCK AND ABLATIONTHERMAL DISC TESTS FOR THE EVALUATION OF
GRAPHITE FOR AEROSPACE APPLICATION

R. A. Meyer

D. E. Niesz (Battelle Memorial Institute): From 8 up to 52 kilowatts input I think way overestimates the difference in graphites. Can you make some comment on what difference you think really exists? When you are putting in more power you are losing a lot more energy.

Meyer: This, of course, is a difficulty of the test, to know exactly how much energy is going into each graphite sample. What has been done, however, is to evaluate a graphite at a power level, relative to the next graphite. Therefore, the test is considered a relative one, as previously mentioned. Some analysis and consideration is being given to this aspect of the test method, for it is very pertinent to relating the test conditions to reentry conditions.

J. G. Crose (Aerospace Corporation): In the across-grain tests, was the plane of the disc in the plane of isotropy for the material?

Meyer: No, the plane of isotropy was not in the plane of the disc.

A TEMPERATURE/STRESS TEST FOR POLYGRAPHITES

H. S. Starrett

J. Jortner (McDonnell Douglas Astronautics Company): How do you tell when your specimen starts to crack?

Starrett: When failure occurs?

Jortner: Yes.

Starrett: Well, there are a variety of ways. For the POCO graphite you saw, it's reasonably obvious, because you get hot chips. But normally you will hear it crack, and you will also see the specimen move. The way this is set up, you can look at it during the run; so you usually see it crack. The other way seems to be the only way that we really have definitely of telling: when it does crack there is a discontinuity, and the thermal gradient shows up immediately, right where the crack occurs.

Jortner: Do you have thermocouples mounted to each specimen?

Starrett: Not every run, but this is the way we've made the temperature measurements without mounting thermocouples to the specimen. Some of these were just run to see if we could crack them.

J. C. Rowley (Los Alamos Scientific Laboratory): I'm curious about the semantic difficulties over thermal shock. "We don't like to call it thermal shock." What's the problem?

Starrett: Well, the problem in calling it thermal shock, as you saw where the heat input in the coupling is a problem, is if you start talking thermal shock, somebody will walk up to you and say, "Will you please rank my graphites for me? Here, I have six. Stick them in your facility and tell me which is the best thermally." Well, this is a very difficult thing to do, because of this coupling problem. And without analysis, I don't think that you can rank the graphites, which is what is usually associated with thermal shock. We hope to use this as an analytical tool, and not as just a little laboratory curiosity.

Rowley: When we developed it, we just called it ZAP. Let everybody make their own conclusions.

Starrett: That's what we hope to avoid!

THERMAL SHOCK RESISTANCE OF EXPERIMENTAL GRAPHITES

R. S. Valachovic and G. T. Yahr

R. A. Meyer (McDonnell Douglas Astronautics Company): I wonder whether, along with the proposed direction, you used fillers which were fired at even lower temperatures, so that you might get even more shrinkage than you probably got with the firings of the fillers you reported.

Valachovic: Well, I'm really not very familiar with the manufacture of these experimental graphites. These were all done by John Napier in the Y-12 plant. We simply ran the thermal shock tests.

G. B. Marrow (Oak Ridge Y-12 Plant): Generally this is true, that the lower the firing temperature of the filler material, with the subsequent high firing at the manufacturing step, or the final consolidation step, the higher the stress resistance or the thermal shock resistance.

C. D. Pears (Southern Research Institute): John [Rowley], I think this paper and our [Starrett's] paper show why we're afraid of ranking, and why we think we are going to have to get the strain analyzer and get an analysis. I also think it's sort of humorous. If we were to rank the materials, which we didn't do, we would have the POCO on the bottom of the list. If you ranked the material, you would have the POCO on the top of the list. So if we were just to rank, we would say it was the worst and you would say it was the best.

Valachovic: It depends on your application.

Pears: I know, and that's why I say the fault of the thinking. You then do things to your material that raises your thermal shock number, and so you are concluding this is a good thing to do to that material. Well, the inference is that if I were to do the same things, on my scale it would drive it down in the other direction, and so when you change something that improved your thermal shock factor, it was going to make mine look worse. So you could change the filler and say, "Ah; that's good," and if I ran it on my tests, I'd say, "Gee, that's good; don't change that filler." We are not materials people. We're mechanics, and it's this kind of problem that we want to stay away from.

G. B. Engle (Gulf General Atomic): Why didn't you use raw coke with a binder? Then I think you would have the maximum amount of matching of the shrinkage of binder and filler.

Valachovic: I'm sure there are things involved in the manufacture of these experimental graphites that I'm not aware of. Why raw coke was not used, I wouldn't have any idea. George Marrow may be able to shed some light on that.

Marrow: We haven't used raw coke. I presume you mean very low-fired coke. I think it was something in the neighborhood of 600 or 800°, where most of the large volume change has already been accommodated to the coking of the material. We want to stay away from the very large things. It's important to understand that we're trying to match the characteristic of the binder and the filler through their thermal history as the material is being processed, and if there is a large mismatch anywhere along there, then you are going to lose the model.

F. C. Weiler (Aerotherm Corporation): Why did you want to develop a graphite that had a high coefficient of thermal expansion? You seem to correlate a high coefficient of thermal expansion with a high shock resistance. I think I'm in agreement with my colleagues in the space field that we're looking for a low coefficient of thermal expansion.

Valachovic: This particular graphite was developed for a special application where that property was desired.

W. S. Scheib (U.S. Atomic Energy Commission): It's to match with the coating that's applied to the graphite.

J. C. Rowley (Los Alamos Scientific Laboratory): Colt [Pears], I couldn't agree with you more. The interesting thing here today, which you probably wouldn't have heard three or four years ago, is four or five people trying to make meaningful thermal stress tests, and use them. I make my check on my ranking, by the way, with a steady state device and a shock device. When I can get agreement on the ranking of those two, I know I have what I want. There are a lot of tricks to this. I wish you luck.

Yahr: I think it's quite easy, Colt, to see how your test gives more weight to thermal expansion than, say, our test. It's simply the fact that you've got more material there that is expanding.

CORRELATION OF EXPERIMENTAL CARBON AND GRAPHITE ABLATION DATA

I. Auerbach, D. F. McVey, and D. D. McBride

W. Chard (Pfizer, Inc.): Did you anneal any of those graphites, especially the CVD infiltrated material; was that annealed prior to testing, or graphitized?

Auerbach: No. Some of the CVD materials were carbonaceous. Others had been heat treated to about 2600°. Some of the AXF-5Q samples had been densified and annealed. Some had been densified and unannealed. This work, incidentally, was done at Battelle. The densified material went up to a density of 2.13 grams/cc, which was a big step from its original density of 1.82.

The statistics indicate that the annealing or the densification didn't do much good. I must be very frank: The sampling is rather small, and perhaps to really get a decisive answer on that, we should rerun that with a larger number of samples.

J. J. Lombardo (NASA Lewis Research Center): I just wanted to ask you, how did you use the statistics? I know $\bar{X}$ and sigma, but what did you compare it to? And since you had a very large difference in the coefficient of variance, some of your high numbers with low variance might be better candidates than some of your lower numbers with very high coefficients. How did you use your statistics?

Auerbach: I depended very largely on our statistician to give me these results!

R. A. Meyer (McDonnell Douglas Astronautics Company): In the interests of having this group assemble next time, I would like to say our data is coming along, and let's continue this at the next meeting. The initial look at this shows on some of our graphites that the initial rate of ablation, that it is microstructurally oriented. That it doesn't seem to be too dependent towards the end. But I don't want to say much more beyond that so far.

Auerbach: We do respect what you say. Just about a year ago we presented a paper at the AIA meeting in which we talked about our observations on microstructure, and its relationship to ablation, and of course many of the remarks which we made there are quite contrary to the remarks which I've made here. We had very good reasons for saying that microstructure was very important to the way the material ablates. Right now I have the feeling that perhaps there are certain statistical factors

that should be taken into consideration before one comes to these conclusions. I think that there is definitely a relationship between microstructure and ablation, but I also think that there are such things as variabilities in the material which somehow mask the microstructure features which influence the ablation.

W. T. Barry (Aerospace Corporation): Essentially you summarize the experience of all the rest of us who have had experience in looking at the ablation of graphite. At the moment they all seem to ablate very much alike, regardless of what their microstructure is; and in spite of what Bob Meyer says about this initial transient, that still can be a facility oriented process. After they go through that first few tenths of a second, they all level out and appear to ablate just the same. This really is a mystery. The heat transfer rate also ought to be affected by the grain size; you should be able to get a difference in roughness; say between POCO and G-90 there should be a tremendous difference, just in the heat transfer. And yet we don't see any effect. There's a mystery here in that somehow we haven't got onto what it's all about.

STRUCTURAL DESIGN CRITERIA AND PROOF TESTINGEFFECTS OF PROOF TESTING AND ANNEALING
ON AXF-5Q GRAPHITE

J. Jortner

G. A. Pinkas (NASA Lewis Research Center): Did you find that your technique enabled you to look at 16 plates and know which ones to throw away? In other words, did you find, for instance, that your 4500 psi tensile load test data was generally confined to one plate so that you could say, "This plate is obviously no good; I'll throw it away"? Or did you find that in one plate you might have a 4500 psi sample and a 9000 psi sample, so you were really not sure, and if you wanted to play it safe you would throw that one away?

Jortner: We tested a number of samples from each plate, and of course the variation within a plate was less than the total variation for the population as a whole. But you couldn't satisfactorily rely on cutting little pieces from each plate, testing them, and then saying the rest of the plate was good. For example, the low value of 4500 psi did come from a plate which had pretty much average properties. So it's a problem.

R. S. Valachovic (Oak Ridge National Laboratory): On your last slide I was very interested in the unloading portion of your compressive specimen. Could you explain a little of the details, how these compression tests were made? Whether you used a compression subjig or a load cell, and give some details? [Editor's note: Figures were subsequently incorporated in the paper to implement the following answer.]

Jortner: Yes. Figure 10 shows the compressive test setup. Figure 11 shows the test specimen.

The strain was measured on most of our samples with a non-averaging Instron extensometer. However, when we saw this seemingly anomalous unloading behavior, we tested a sample using strain gages and found the same S-shaped unloading. Possibly in going up to loads where the tangent modulus begins to decrease significantly (see Fig. 9), we are getting some buckling, and that may in some way be related to the way the specimen unloads. However, that's a rather remote possibility, because you would expect the plane of buckling to be random with respect to the strain measuring device. The fact that we saw the same stress-strain behavior on all of the samples leads me to believe that buckling was not responsible for the unloading behavior, because, depending on where one mounted the instrument, one would have measured different strains if the specimen were buckling.

SHORT NOTES

A TECHNIQUE FOR EVALUATING THERMAL STRESS RESISTANCE

D. E. Niesz and W. H. Duckworth

J. C. Rowley (Los Alamos Scientific Laboratory): How did Gene ever convince the materials people to test in a nonhomogeneous stress field?

Niesz: Well, we have few other choices in order to stay in the temperature range where we do not get plastic flow. A question which we hope to answer for graphite is whether the stress state around the notch affects the fracture mode.

Rowley: Why did he put a radius in the end of that notch? I tried to talk him into using a flat-bottom notch. It's easier to do a finite analysis method, and I don't think it really matters from a materials standpoint, or do people argue that?

Niesz: Well, of course you get a much higher stress concentration with the square-bottom notch, but your finite analysis essentially blows up if you have a discontinuity whose dimensions you can't describe. You can't get a perfectly square notch, and that's the reason we prefer to use one with a radius. We can define the geometry. You can't do that for a square notch. At least that's Gene's feeling!

Rowley: I'm still not convinced! I think the finite analysis blows up in both cases, because he's still using a linear model, and we know the stresses and strains are high, and we know they don't bear any particular resemblance to a stress-strain curve, which you get from a homogeneous test. What's the alternative? This is really a difficult testing area, but have you tried any different kinds of notches to see if it really matters? My suspicion is that it doesn't.

Niesz: We haven't done a lot of testing with different notches. We have put what looked like square-bottom notches and round-bottom notches in, and there doesn't seem to be much effect in terms of response. We really haven't done enough to say whether there is a significant effect. One of the things we want to look at is just how well the analysis tells us stress-concentration factor, and we are considering taking a notch specimen and looking at it under no gradient and with a thermal gradient by holography to see what the discontinuity and strain are at the notch.

Rowley: I think it would be very revealing if you could get somebody to put a microscope in there and watch. I think you would be terribly disturbed, because you will see the whole area, I suspect, go very nondense. You are going to get a whole dedensification of that highly stressed area, and flaws grow, and cracks extend, and the whole thing.

As a materials person, this must be very disturbing. You have introduced this drastic geometric and material disturbing influence, and yet you're willing to go ahead and do a test on this. How did Gene convince you?

Niesz: I don't think he convinced me. We just did it to see whether it would work, and we really don't know how well we can analyze it at this point. But we do know that you might worry, for example, that because of the way graphite fractures, you get microcracking, or it relieves the stress concentration somewhat, and you don't get fracture; but then indeed you do get fracture, and it seems to behave much like it does in a fracture mode of any kind. You start getting microcracks and then it fails. I think we need some sort of controlled evaluation of what happens--where our material responds to thermal stress and how it fractures.

You can also do this by going to about ten times the heat flux we're using and cooling the outer surface to keep the temperature down to something below 6000 amp. or so, which is about where it goes to. You can also do it in a transient condition, and I think several papers are going to be given on that at this conference.

M. P. Whittaker (Great Lakes Carbon Company): What is the sample size?

Niesz: The sample size that we use most is about an inch ID by about 2-1/2 to 3 inches OD, and an inch high.

Whittaker: Can you go to larger size?

Niesz: We wouldn't want to go too much higher in diameter, but length, we can go up to possibly 4 inches.

APPLICATION OF MONTE CARLO TECHNIQUES IN STRUCTURAL ANALYSIS FOR A NUCLEAR ROCKET

P. P. Zemanick (presented by R. Bolton)

J. C. Rowley (Los Alamos Scientific Laboratory): You say you don't need material properties. You just get a big statistical problem and throw it in the computer.

Bolton: This only determines the load, not the material properties.

STATISTICAL CONSIDERATIONS ON THE STRENGTH OF NUCLEAR
GRAPHITES: CHARACTERIZATION, IRRADIATION, AND DESIGN

V. Lungagnani and R. Krefeld

G. T. Yahr (Oak Ridge National Laboratory): Did the size effect that you saw correlate with the value of m that you calculated from the distribution? In other words, did you have as much of a size effect as the Weibull distribution would predict?

Lungagnani: In which experiment are you speaking of? Of the measured variation of the strength as the function of the volume. That was just an experiment. That is not systematic. In other words, I cannot say what would be the behavior for very big blocks or for very small specimens. What we have seen is that for specimens of reasonable size, for reasonable laboratory testing, the variation is quite high, and you have seen it? Is that your question?

Yahr: The question really is if you take, say, your smallest volume, or your next smallest volume, where you got a good Weibull distribution, and use that distribution to calculate the main strength of your largest volume, how do the results compare?

Lungagnani: We didn't, but I feel, with the smallest volume, that is not represented by a Weibull distribution. I can show you, if you like, the photomicrograph, of how this material looks, in some cases. And you can understand very clearly that a specimen 4 mm. in diameter does not have any chance to be an average on the structural inhomogeneity of the material.

Yahr: But you could use your next smallest volume and compare your results there with the largest volume; right?

Lungagnani: Yes, that could be.

G. B. Engle (Gulf General Atomic): At the neutron dose to which these samples were taken, I note that Gilsonite graphite at that temperature will just be approaching its maximum densification with dose, and I believe the Dragon data on a similar type of graphite would show that the modulus would be increasing at this particular dose. Would you care to comment on the reduction of strength accompanied by the increase in modulus?

Lungagnani: It is O.K., for a generalization of the graphite behavior under irradiation, that as soon as the material is densified, its modulus is increased, and its strength, too. That implies that the irradiation damage is the same for all graphitic materials in all temperature ranges. What we have seen, and I did that work with Dragon in France, together, is that the evolution of the Young's modulus on the Gilso material is quite peculiar. The data of France for the Young's modulus show that with increasing neutron dose the modulus for normal (needle-coke) graphite reaches a peak, then decreases, then increases again to a higher

maximum. For Gilso graphite, however, the modulus reaches a single maximum, begins to decrease at a neutron dose of the order of the dose that produces the first peak in needle-coke graphite, then continues to decrease. So you can argue that these represent further densification, and then you can expect a higher strength. But I don't have an explanation for this behavior, and that way I don't have an explanation for the non-densification, and then I don't have an explanation for the loss in strength that is there.

So of course, it's 1200° irradiation, so I cannot disclose some side effects; like an inleakage of oxygen, or some internal corrosion, or something like that. You have to realize that it is a destructive test. It's not too easy to repeat it.

E. L. Foster (Battelle Memorial Institute): I think this bears on the same question, but I wanted a little different statement of it. In the population of the samples that you tested after irradiation, and showed the differentiation between two distributions, you said the one showed a dramatic increase in strength. Would you associate that most with change in modulus, or change in strain to failure?

Lungagnani: That is a good question, but I don't have the data with me. I decided to give this lecture on the spot. I can write to you and let you know that. [Editor's note: The technical content of a letter from R. Krefeld to E. L. Foster in answer to the above question is reproduced below (from a copy provided by V. Lungagnani)]:

"Fast neutron irradiation can cause an increase of the strength of the graphite. This increase was, according to our observations, always associated with an increase in modulus. Total strain to fracture was decreasing due to neutron-induced embrittlement. The order of magnitude of these effects is strongly dependent on the irradiation temperature, besides the total fluence. The higher the irradiation temperature the smaller are these effects, because of thermal annealing."

FABRICATION OF ISOTROPIC FIBROUS CARBON-CARBON COMPOSITES

J. L. Cook and F. Lambdin

W. Chard (Pfizer, Inc.): In your studies, have you noticed any breaking up of the fibers during compaction? It's been my experience in working with hot isostatic compaction of various composite materials, that if you don't have a very plastic matrix, the fibers will break up so that your L/D ratio completely shifts during the compaction process. Also, do you have any data on flexure strength of the material?

Cook: I think probably you might expect some cracking up of the fibers here. You notice I didn't make any play to say that we were building a structurally efficient composite, and therefore it might not be

that important to us that we lose some of the L/D of the fibers. We are in a little better situation to avoid that, since our compaction is all done warm and continually increasing in temperature, so that we have a fluid matrix during the major portion of the compaction. The worst possible point to me is going through that Wiley mill. But they seem to come out, as you saw the agglomerates. This is typical of what we see.

On the second question: Yes, there are some data available, and Irv Auerbach perhaps will say a little bit about it in his talk tomorrow. One number that I could quote you that we've obtained on the material at a density of 1.8 is about 5000 psi in flexure at room temperature.

Chard: The lower strength of the material could possibly account for some of the improved thermal shock characteristics of the material. In other words it could accommodate some microcracking, etc., during thermal testing.

Cook: Let's put it this way: At a given strength of material, perhaps the combination of other properties would be more important, as far as I'm concerned. That is the conductivity and the strain to failure. I'm not accustomed to thinking of reducing the strength to improve the thermal shock resistance. I would say this: Terry Yahr and I have checked out some density variations of this material, where the porosity is quite high. And indeed you can get significant shock resistance utilizing the porosity as a crack deterrent. I don't think that's an objective of a significant improvement in material for ablation and thermal shock resistance. I think you want to go for optimum homogeneity, fine grain size, and essentially 100% of theoretical density in the material, if you can get that with thermal shock resistance. We can live off of that porosity for a while, but, you know, we're eventually going to be up against it on our material properties.

W. G. Bradshaw (Lockheed Missiles and Space Company): What kind of fibers did you use? Also, if you're processing only to 2000°F, are you going to encounter some dimensional instabilities when you are operating at 6000°F?

Cook: Could we take a look at that first slide again? The fiber type that we used was a 2-micron-diameter rayon fiber, graphitized to 2000 to 2100°C, not F, and those fibers are a development of the Y-12 Plant. They're smaller in diameter than most readily available fibers. And we chose that diameter to enhance our ability to get a finer grain size in these materials; the finer fibers.

The processing, you will notice, was indicated here to 2000°C during the processing. On final graphitization it would go to 3000°, very often, centigrade. We haven't observed a lot of shrinkage change on going from 2000° to 3000°, however.

APPENDICES

APPENDIX A

PROGRAM

CONFERENCE ON "CONTINUUM ASPECTS OF GRAPHITE DESIGN"

Riverside Motor Lodge
Gatlinburg, Tennessee
November 9-12, 1970

Sunday, November 8, 1970

5:00 P.M. — 9:00 P.M. Registration
6:00 P.M. "Smoker"

Monday, November 9, 1970

INTRODUCTION

Opening Remarks:

8:50 *D. B. Trauger*, Associate Director, Oak Ridge National Laboratory
9:05 *J. E. Fox*, Co-Chairman Graphite Coordination Working Group
9:15 *W. L. Greenstreet*, Oak Ridge National Laboratory

KEYNOTE LECTURE

9:20 Description of Design Requirements for Graphite Components.
J. C. Rowley, Los Alamos Scientific Laboratory
10:20 Intermission

SESSION 1. MATERIAL CHARACTERIZATION

10:40 Chairman — *E. J. Seldin*, Union Carbide Corp.
10:50 Correlation of Properties of Manufactured Graphites. *P. E. Armstrong*,
Los Alamos Scientific Laboratory
11:10 Mechanical and Thermal Properties of Airco Speer Grades for Nuclear
and Aerospace Applications. *L. F. Simbeck*, Air Reduction Co., Inc.
11:30 Mechanical Behavior of Artificial Graphites as Portrayed by
Uniaxial Tests. *W. L. Greenstreet*, *J. E. Smith*, *G. T. Yahr*,
and *R. S. Valachovic*, Oak Ridge National Laboratory
11:50 Mechanical and Thermal Properties of Glass-Like Carbons.
W. G. Bradshaw, Lockheed Missiles and Space Co.
12:10 Lunch

SESSION 2. MATERIAL CHARACTERIZATION (Continued)

- 1:40 Dynamic Biaxial and Elevated-Temperature Properties of ATJ-S Graphite. *S. G. Babcock, S. J. Green, P. A. Hochstein, and J. A. Gum*, General Motors Corp.
- 2:00 Material-Design Concepts for Filament-Wound Graphite — Graphite Heatshields. *T. R. Guess* and *C. W. Bert*, Sandia Laboratories
- 2:20 The Microstructural Evaluation of Graphites for Their Erosion and Ablation Characteristics in Aerospace Applications. *R. A. Meyer, K. M. Kratsch, and R. B. Dirling, Jr.*, McDonnell Douglas Astronautics Co.
- 2:40 Characterization of Production-Grade H-327 Graphite for HTGR Design. *H. R. W. Cobb, S. J. S. Parry, and G. B. Engle*, Gulf-General Atomic, Inc.
- 3:00 Intermission
- 3:20 Characterization of Several Typical Polygraphites with Some Convergence on Solid Billet ATJ-S. *C. D. Pears*, Southern Research Institute
- 3:40 Microstructural Analysis to Determine Elastic Material Properties of a Filament Wound Graphite Composite Having Orthotropic Constituents. *J. H. Biffle*, Sandia Laboratories
- 4:00 Holographic Interferometric Studies of Carbon Composite Cylinders. *O. J. Burchett, C. G. Murphy, and C. W. Matthews*, Sandia Laboratories
- 4:20 Ultrasonic Characterization and Computerized Analysis of Graphite Billets. *R. A. Baker, D. E. Bishop, and G. C. Stoker*, Sandia Laboratories
- 4:40 Adjourn
- 6:00 Graphite Mixer

Tuesday, November 10, 1970

SESSION 3. CONSTITUTIVE EQUATIONS

- 8:50 Chairman — *C. E. Pugh*, Oak Ridge National Laboratory
- 9:00 Mathematical Representation of Mechanical Behavior of Nonlinear Inelastic Materials. *E. T. Onat*, Yale University
- 10:00 An Elastic-Plastic Continuum Theory for Artificial Graphite. *W. L. Greenstreet*, Oak Ridge National Laboratory
- 10:20 Intermission
- 10:40 Stress-Strain Properties of Grade ATJ Graphite Under Combined Stresses. *T. Weng*, Union Carbide Corp.
- 11:00 Thermomechanics of Rate-Independent Materials with Memory. *R. D. Snyder*, West Virginia University

- 11:20 Possibilities for Application of Higher-Order Continuum Theories.
F. Essenburg and W. G. Gottenberg, University of Colorado
- 11:40 A Formulation of the Mechanical Behavior of ATJ Graphite at High Temperature. *T. B. McDonough, General Electric Co.*
- 12:00 Lunch

SESSION 4

A. CONSTITUTIVE EQUATIONS (Continued)

- 1:30 Viscoelastic Representation of Anisotropic Mechanical Behavior of Graphite at Elevated Temperatures. *C. E. Pugh and J. P. Callahan, Oak Ridge National Laboratory*
- 1:50 A Viscoelastic Representation of the Behavior of Graphite Under the Influence of Neutron Irradiation. *S. J. Chang, Oak Ridge National Laboratory*

B. STRUCTURAL ANALYSIS — NUCLEAR

- 2:10 Chairman — *J. M. Corum, Oak Ridge National Laboratory*
- 2:20 Viscoelastic Response of Graphitic Materials in Irradiation Environments. *T. Y. Chang and Y. R. Rashid, Gulf General Atomic, Inc.*
- 2:40 Analysis of Spherical Coatings on Nuclear Fuel Particles. *J. W. Prados and H. C. Roland, University of Tennessee*
- 3:00 Intermission
- 3:20 A Model for Thermal and Radiation Stress and Strain in Viscoelastic, Orthotropic Cylinders, and Its Application to Borated-Graphite Absorbers. *D. W. Stevens and O. M. Stansfield, Gulf General Atomic, Inc.*
- 3:40 Structure Design of Nuclear Rocket Fuel Elements. *P. P. Zemanick, Westinghouse Astronuclear Laboratory*
- 4:00 The NERVA Nuclear Subsystem Friction-Damped Lateral Support System. *R. Bolton, Westinghouse Astronuclear Laboratory*
- 4:20 Steel-to-Graphite Connections Analyzed by Finite Element Techniques for Use in NERVA Nozzle Extension. *W. de la Garza, Aerojet Nuclear Systems Co.*
- 4:40 SHORT NOTES — PART 1
- Chairman — *C. R. Kennedy, Oak Ridge National Laboratory*
- 5:30 Adjourn

Wednesday, November 11, 1970

SESSION 5. STRUCTURAL ANALYSIS — SPACE

- 8:50 Chairman — *T. McKinley*, Aerospace Corp.
- 9:00 Anisotropic Elastic-Plastic Stress Analysis of Axisymmetric Solids.
F. C. Weiler, Aerotherm Corp.
- 9:20 ASAAS — Asymmetric Stress Analysis of Axisymmetric Solids.
J. G. Crose, Aerospace Corp.
- 9:40 Thermal/Structural Analysis of Designs Using Graphitic Materials.
R. D. Teter, Lockheed Missiles and Space Co.
- 10:00 Structural Analysis of Carbon and Graphite Filament Wound Heatshields
for Reentry Heating Environment. *J. T. Schamaun*, Sandia Laboratories
- 10:20 A Thermostructural Sensitivity Analysis for Graphitic Nose Tips.
R. J. Michalak, *T. B. McDonough*, and *W. Connell*, General Electric Co.
- 10:40 Intermission
- 11:00 SHORT NOTES — PART 2
Chairman — *C. R. Kennedy*, Oak Ridge National Laboratory
- 12:00 Lunch

SESSION 6. FRACTURE

- 1:30 Chairman — *Lt. J. R. Koenig*, Air Force Materials Laboratory
- 1:40 The Deformation and Fracture Mechanisms in Polycrystalline Graphites.
M. C. Smith, Los Alamos Scientific Laboratory
- 2:00 Observations of Fracture Phenomena in Polygraphites and the
Resulting Arguments of Fracture Mode and Criteria. *C. D. Pears*,
Southern Research Institute
- 2:20 A Simplified Approach to Predicting the Onset of Fracture in
Brittle Materials. *R. W. Andrae*, Los Alamos Scientific Laboratory
- 2:40 The Mechanical Properties of Graphites and Their Relation to
a Physically Based Statistical Theory of Fracture. *J. D. Buch*,
Aerospace Corp.
- 3:00 Intermission
- 3:20 Chairman — *W. P. Eatherly*, Oak Ridge National Laboratory
- 3:30 Biaxial Mechanical Properties of AXF-5Q Graphite to 4000°F.
J. Jortner, McDonnell Douglas Astronautics Co.
- 3:50 Geometrical Parameters Affecting the Fracture Toughness of
Graphite. *G. T. Yahr* and *R. S. Valachovic*, Oak Ridge National
Laboratory
- 4:10 Fatigue of Graphite. *G. K. Mahajan* and *L. J. Broutman*, Illinois
Institute of Technology

SESSION 6. FRACTURE (Continued)

- 4:30 Application of Weibull Statistical Theory to the Strength of Reactor Graphite. *R. J. Price* and *H. R. W. Cobb*, Gulf General Atomic, Inc.
- 4:50 Adjourn
- 7:00 Cook-out

Thursday, November 12, 1970

SESSION 7. THERMAL SHOCK AND ABLATION

- 8:50 Chairmen — *K. R. King*, TRW Systems Group, and *G. T. Yahr*, Oak Ridge National Laboratory
- 9:00 Transient Thermal Stresses in Graphite. *K. R. King* and *J. R. Bohn*, TRW Systems Group
- 9:20 Thermal Disc Tests for the Evaluation of Graphite for Aerospace Application. *R. A. Meyer*, McDonnell Douglas Astronautics Co.
- 9:40 A Temperature/Stress Test for Polygraphites. *H. S. Starrett*, Southern Research Institute
- 10:00 Thermal Shock Resistance of Experimental Graphites. *R. S. Valachovic* and *G. T. Yahr*, Oak Ridge National Laboratory, and *J. M. Napier*, Union Carbide Corp. Y-12 Plant
- 10:20 Correlation of Experimental Carbon and Graphite Ablation Data. *I. Auerbach*, *D. F. McVey*, and *D. D. McBride*, Sandia Laboratories
- 10:40 Intermission

SESSION 8. STRUCTURAL DESIGN CRITERIA AND PROOF TESTING

- 11:00 Chairman — *G. B. Marrow*, Union Carbide Corp. Y-12 Plant
- 11:10 NERVA Design Criteria and Application for Brittle Materials. *G. J. DeSalvo*, Westinghouse Astronuclear Laboratory, and *J. J. Lombardo*, Space Nuclear Propulsion Office — Cleveland
- 11:30 Effects of Proof Testing Annealing on AXF-5Q Graphite. *J. Jortner*, McDonnell Douglas Astronautics Co.
- 11:50 Sandia MTV/Carbon-Carbon Program. *H. W. Schmitt*, Sandia Laboratories
- 12:10 Adjourn

Meeting Chairman: *W. L. Greenstreet*

Meeting Coordinators: *J. P. Callahan*
R. S. Valachovic
G. T. Yahr

APPENDIX B
SESSION ORGANIZERS

<u>Session</u>	<u>Organizers</u>
Material Characterization	<i>E. J. Seldin</i> (Union Carbide Corp.)
Constitutive Equations	<i>C. E. Pugh</i> (Oak Ridge National Laboratory)
Structural Analysis	<i>J. M. Corum</i> (Oak Ridge National Laboratory) <i>T. McKinley</i> (Aerospace Corp.)
Fracture	<i>W. P. Eatherly</i> (Oak Ridge National Laboratory) <i>Lt. J. R. Koenig</i> (Air Force Materials Laboratory)
Thermal Shock and Ablation	<i>G. T. Yahr</i> (Oak Ridge National Laboratory) <i>K. R. King</i> (TRW Systems, Inc.)
Structural Design Criteria and Proof Testing	<i>W. L. Greenstreet</i> (Oak Ridge National Laboratory)
Short Notes	<i>J. P. Callahan</i> (Oak Ridge National Laboratory)

APPENDIX C

ATTENDEES

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