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WANL-TME-870
August 11, 1964

MASTER

REACTOR NON-FUEL MATERIALS PROGRAM
(1964 AND 1965)

Structural Materials and Processes
Materials Department

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Subject: WANL-TME-870
REACTOR NON-FUEL MATERIALS PROGRAM
(1964 and 1965)

Gentlemen:

The subject report describes the 1964 accomplishments of the Structural Materials and Processes Group of the Materials Department on Project 8910. It also proposes a work program for C.Y. 1965 in this area under Project 2800.

Yours very truly,

H. F. Faught

H. F. Faught
Program Manager
NERVA Nuclear Sub System

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REACTOR NON-FUEL MATERIALS PROGRAM
(1964 AND 1965)

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August 11, 1964

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INTRODUCTION

Both the CY 1964 and proposed CY 1965 Work Programs are presented in the following with the same basic outline as used for presentation of the CY 1964 proposal in TME-426:

- A. Vendor Liaison, Vendor Evaluation and Consultation
- B. Materials Evaluation, Application and Behavior
- C. Fabrication Development
- D. Irradiation Effects
- E. Specifications

The CY 1965 Program has been planned for a level of activity of 15 engineers, 9 technicians, and \$120,000 for materials and services. The approximate manpower distribution for this plan is indicated in Appendix "A". The program is a reduction from 225 to 180 engineering man-months and from \$150,000 to \$120,000 for materials and services.

Most of the details of the CY 1964 Program Accomplishments have not been included here, for they are available in the monthly and quarterly progress reports and in topical reports. The topical reports, both those issued to date and those to be issued later in CY 1964, are listed as Appendix "B". The day-to-day contacts with personnel in other departments, such as Mechanical Design, Manufacturing, Quality Control, Reactor Analysis, Control Development, and Purchasing, and with vendors provide the basis for formulating the proposed CY 1965 Work Program. This program has been reviewed with the management of Mechanical Design, Reactor Analysis, Instrumentation and Control, and Engineering Mechanics to assure that it meets their anticipated need for materials support of the NERVA project and to avoid duplication of effort.

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CY 1964 ACCOMPLISHMENTS

A. Vendor Liaison, Vendor Evaluation and Consultation

Much of the day-to-day activity on Project NE 8910 consisted of vendor liaison, vendor evaluation and consultation in close support of the design effort, hardware procurement and reactor assembly. As a result, many of the materials and process specifications were revised to better reflect design needs and vendor capabilities. Particular attention was paid to the pyrolytic graphite vendors because of the NERVA need for improving the state-of-the-art. Although many pyrolytic graphite problems remain, progress has been made in increasing density, decreasing delaminations, improving dimensional control, increasing the extent of regenerative microstructure and improving uniformity of product. Other NERVA materials problems necessitated liaison with suppliers of structural graphite, beryllium, titanium alloy forgings, tie rods, aluminum alloys, nozzle end seals, aluminum forgings, aluminum weldments, metal finishing and others.

Consultation services have been performed as a continuing effort throughout the contract year. Two particular functions have been to provide metallurgical advice for the evaluation and selection of materials for components and to provide general materials properties data to other departments in response to their requests. Frequent consultations, including trouble shooting, were held with WANL staff members in various departments. These dealt with a broad spectrum of materials problems, both present and anticipated. Manufacturing, Purchasing, Quality Control and Design were among the areas in which information was most often sought.

More specifically, close technical coordination and cooperative testing resulted in improved raw materials, irradiation resistant instrumentation devices and acceptance of ultrasonic inspection for the first time by beryllium suppliers. Assistance in fabrication development made it possible to secure cluster plates made from gadolinium-bearing stainless steel. Close coordination improved the upset forging techniques and produced tie rods satisfactory for the NERVA needs. These efforts also made it possible to improve the procedure for cementing tiles to filler strips, welding and heat treating the core barrel and control drum housings, and to properly assemble the flow screens and instrumentation devices. Studies

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in the Materials Department on physical and mechanical properties and quality of raw material followed by close vendor liaison made showed that it is possible to secure improved raw material for inner reflector cylinders, graphite tubing free of x-ray defects for structural modules, higher quality pyrolytic graphite components, beryllium that can pass ultrasonic inspection standards, and improved candidate raw materials for support blocks. After a number of instrumentation devices failed in cryogenic irradiation tests, improved materials were recommended and modified designs were suggested to the vendors, and it was possible to produce new instrumentation devices which have successfully passed cryogenic irradiation tests. Trouble-shooting efforts on the causes for nozzle-end seal ring failures have resulted in approval of a new vendor, improved fabrication methods, and raw materials selection.

Substantial manpower effort has been required on a continuing basis for consulting with other groups, coordinating component tests to prove material behavior, and on trouble shooting investigations to solve problems that periodically appear. An intensive review of the compatibility of component environment and material properties was conducted to recommend materials for advance designs and changes in existing reactor designs. Studies of material behavior were conducted to determine the relative risk of hydrogen embrittlement of stressed and unstressed titanium in elevated temperature static hydrogen atmospheres. Irradiation effects which could impair the accuracy of cryogenic temperature measurements were called to the attention of the instrumentation group.

The detrimental effects of irradiation on the high-temperature creep properties of structural alloys was also delineated for design groups. Data on material behavior enabling the stress analysis group to calculate high-strain, low-cycle fatigue life was supplied. Since much of this information was based on literature data and extrapolations of material behavior, test programs must be designed in the future to fill the information gaps which exist. Discussions of materials behavior and materials recommendations were presented in design review meetings with the customer. Metallurgical investigations were required to explain failures of the nozzle end seal, the fatigue failure of non-specification grade titanium bolts, leakage in instrumentation lead seals, excessive permeability in graphite cylinders, and for coating failures on non-fueled structural components being tested at elevated temperatures.

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Additional metallurgical trouble-shooting was conducted on x-ray defects observed in large graphite cylinders. Materials evaluation indicated that no significant reduction in mechanical strength could be observed and, as a result, new acceptable standards were established for x-ray-detected high-density defects. Since materials behavior is an integral part of the response of components to a test environment, it was frequently necessary to coordinate component testing to properly evaluate the material use and component design. A typical example can be illustrated by the risk of brittle failure of molybdenum support cones in a cold-flow test where the temperature goes far below the normal steady-state temperature in operation. The transition temperature for ductile to brittle failure and the strain rate sensitivity when coordinated with design conditions then dictated the conditions of a component test which was run and which subsequently established the cones as being satisfactory for their environment. Numerous other component tests were suggested for specific components in the test program to insure the safety of NRX A-1 and 2. Considerable consulting effort also went into the preparation of NRX-A2 test specifications and the delineation of the relative importance of selected post-mortem tests.

B. Materials Evaluation, Application and Behavior

1. Materials Data Sheets

Data acquisition has been primarily guided by the requirements outlined on the Materials Data Worksheets, which relate NERVA Materials, their applications, their requirements and urgency of handbook-type data. Special emphasis was given to the graphites because of their relatively high priority and lack of available information. Although the bulk of the data acquired thus far has been obtained from the literature and suppliers, increasing amounts are being determined experimentally. For example, presently there are no significant tensile properties data available for TZM molybdenum in the temperature range from -250°C (-420°F) to 650°C (1200°F). In view of the use of this material for support cones, more information in this temperature range is required. Tensile and notched tensile tests and metallographic examinations of the fractures are underway.

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Many of the existing data sheets have been transcribed onto a special format and properly referenced as WANL's contribution to the new edition of the NERVA Materials Properties Data Book. A total of 151 sheets representing 16 properties and 31 alloys or grades of graphite have been prepared to date. They include physical and mechanical properties as a function of temperature of 2024, 7075, 2219 and 6061 grades of aluminum; high-temperature alloys, Inconel 718, Inconel X-750, Discaloy, W-545, Rene 41, D-979, A-286, and Incoloy 901; beryllium; A-110-AT titanium alloy; stainless steel grades 304 and 310; Ni-SPAN-C 902; and graphite grade ATJ.

2. Graphite Evaluation Studies

a. Inner Reflector Cylinders

An evaluation has been made of the mechanical and physical properties of extruded H4LM graphite which is used for the inner reflector cylinder. Consideration had been given to the use of a molded grade, ATL, and it too was evaluated. Because of the large difference in the radiographs of these cylinders, this program was increased in scope to include all of the 15 cylinders at WANL in support of a manufacturing effort to direct the best cylinders to the hot-test reactors.

The major conclusion reached in this evaluation is that extruded H4LM graphite is preferred to molded MHLM or ATL graphite grades. Test results, however, show a large lot-to-lot variation which is indicative of the minimal quality control effort used in the manufacture of these low-cost materials. Cylinders of one lot were noted to have an unusually large number of radiographic indications as well as lower strength. No correlation, however, was noted between the strength of a specimen and the radiographic indications it contained. Apparently some of these indications are due to impurity concentrations or areas containing pockets of low and high-density graphite which have little or no influence on the strength of the test section. Methods for more positive identification of these radiographic indications are necessary if proper evaluation of the graphite is to be made. Experimental results show that the compression strength of silica-impregnated H4LM graphite increases approximately 20%, but there is little or no change in the tensile behavior.

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As a result of this evaluation and negotiations with the supplier, extruded H4LM cylinders may be obtained with guaranteed mechanical properties (at an increase in price) as follows:

<u>Property</u>	<u>Average Strength psi, Min.</u>	<u>Minimum Value psi</u>
Tensile - Axial	1800	1500
Tangential	1900	1700
Flexure -Axial	2600	2400
Tangential	None	
Compressive - Axial	--	5000

A program was initiated to scale-up several grades of graphite which could be used to replace H4LM. In order to make use of these high-strength, low-permeability graphites, the cylinder will have to be redesigned to use an assembly of stacked rings or full-length staves, unless equipment can be found to handle full-sized integral barrels so the process may be tried and evaluated.

b. Structural Graphites for NRX-B

Prior to the redirection of the NERVA program, the assigned task was to provide the NRX-B design effort with reliable data on the high-strength graphite grades suitable for structural uses in the reactor. Early contacts were established with the graphite manufacturers to obtain such data and investigate the graphite grades in advance development. The published literature was reviewed to obtain data for the off-the-shelf grades.

Investigation of the mechanical properties of ATJ graphite was begun to supplement the data obtained from the literature. Initial work consisted of studying the flexural behavior of ATJ to define the usefulness of the flexure tests for the general evaluation of materials. All tests were limited to room temperature. Tensile tests were initiated

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only after a careful selection of two promising specimen designs. Initial testing was limited to Graphitite-G graphite at room temperature. With the availability of additional equipment, tests at high temperatures were undertaken in inert atmosphere on the Graphitite-G and ATJ graphites.

Dense (1.88 g/cc), isostatically molded graphite, grade PO-3 was evaluated by tensile, flexural, and compression tests at room temperature. PO-3 excelled ATJ and ZTA graphites by 25 to 100%. The average thermal expansion coefficient of PO-3 between room temperature and 1800 °C (3270 °F) is $2.76 \times 10^{-6}/^{\circ}\text{F}$ and no permanent dimensional changes occurred. This material has been selected for improved plunger pins and is being further evaluated for seal segments, support blocks, and filler strips.

A survey of extruded graphite grades, both off-the-shelf and in-advance-development, was conducted to determine the optimum material for the NRX-B module tube. After the advanced program's termination a report was issued to summarize this activity, and to present its findings for any future work along these lines. A comprehensive survey of the extruded graphites possessing uniformly high thermal expansion was carried out. Another survey included the molded high-expansion graphites. Generally, the average thermal expansion coefficient was found to be somewhat less than that of the NbC coatings. All of these graphites developed excellent adherence of the deposited coatings. It was felt that the availability of structural graphite for the high-reliability, coated applications would pose no future problems. Initial evaluation of A-122-S showed its thermal expansion in the with-grain direction to be $3.5 \times 10^{-6}/^{\circ}\text{F}$ whereas currently used ATJ shows $2.3 \times 10^{-6}/^{\circ}\text{F}$. For later NERVA reactors this grade should be considered, providing adequate quality control is achieved to minimize macroscopic defects.

Preliminary investigation of the fiber-base graphites was made on the following types of material:

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<u>Grade</u>	<u>Base</u>	<u>Final Processing Temperature</u>	<u>Density</u>
PT-0111	Laminated graphite cloth	2760 °C (5000 °F)	1.10 g/cc
PT-0156	Same with additional binder	816 °C (1500 °F)	1.30 g/cc
PT-0156/2800	Same with additional binder	2760 °C (5000 °F)	1.36 g/cc
PT-0154	Macerated graphite cloth	2760 °C (5000 °F)	1.20 g/cc
PT-0154	Same with additional binder	816 °C (1500 °F)	1.50 g/cc

At room temperature their strength-to-weight properties are comparable to conventional graphites of greater density, and at 2482 °C (4500 °F) these novel graphites have some of the highest strength-to-weight ratios ever reported. The thermal conductivity of fiber-base graphites measured at room temperature is considerably lower (7-27 BTU ft/ft² Hr °F as compared to 65-80 BTU ft/ft² hr °F for ATJ) than those of regular molded and extruded graphites, suggesting that fiber-base graphites may function as moderate thermal insulators. Also, niobium carbide coatings adhere well to PT-0111, which was selected as the structural material for the NRX-B module tube. A summary report is being prepared.

c. Pyrolytic Graphite Materials

Pyrolytic graphite components of each type made by each manufacturer have been evaluated, both in the as-deposited and as-machined conditions. The properties evaluated have consisted mainly of density, macrostructural appearance, dimensional uniformity, microstructure, chemical composition, and thermal conductivity. Across-grain thermal conductivity data were obtained from Southern Research Institute on tubes as a function of mean temperature from 260 °C (500 °F) to 1760 °C (3200 °F), representing the extremes in microstructure (i.e., slightly renucleated and highly renucleated). Pyrolytic graphite plate material was evaluated representing different furnace lots and different vertical positions within lots from each of three manufacturers. Examination of the product of one manufacturer

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showed it to be consistently and uniformly renucleated to a high degree, and contained no delaminations. The other two manufacturers' products were insufficiently renucleated and contained microdelaminations which were usually restricted within single growth cones. Severe thermal gradients were imposed on plate samples from the three sources to determine the effect of microstructure and the number of thermal cycles. The gradients were generated by placing one surface on each sample in contact with a graphite resistance heater and heating it to approximately 2150 °C (3902 °F) in about one minute. The temperature of the opposite surface was optically measured as a function of time. Visual examination of the cycled samples revealed no macro-delaminations. However, these results are insufficient to permit conclusions regarding the effect of micro-structure on the thermal shock resistance of pyrolytic graphite.

Several pyrolytic graphite products were evaluated as improvements to the conventionally made vapor-deposited pyrolytic graphite components as noted hereafter.

1) As-deposited insulating sleeves and cups, made by a proprietary plasma-arc spraying process, represented an attempt to obtain components having a large wall-thickness-to-radius ratio, deposited to size and thus avoiding machining which usually exposes delaminations. The evaluation consisted of visual, dimensional, and microstructural examinations, and across-grain thermal conductivity at 1093 °C (2000 °F) and 1643 °C (3000 °F). Basic problems with this plasma-arc spraying process appear to be obtaining a heavy enough deposit and controlling the microstructure, although the process has potential as reported.

2) The bulk density, true density, flexural strength, compressive strength, Young's modulus in compression, coefficient of thermal expansion, and microstructure of isotropic pyrolytic graphite foam were determined. The material consists of a pyrolytic graphite coating on a graphite substrate. Although interesting, the materials do not seem to have application in the present NERVA design.

3) The evaluation of Grafoil-100 continued in an effort to replace vapor-deposited pyrolytic graphite components with laminated pyrolytic graphite foil components. It included determination of room temperature density, compressibility, tensile strength,

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thermal conductivity and chemical composition. The spectrographic analysis indicated an extremely high level of impurities, undoubtedly from the bonding agent, as the foil itself is very pure. These Grafoil-100 products appear to have a great potential for improving quality and reliability of pyrolytic graphite components. Samples of more pure material are being examined.

d. Niobium Carbide Coating Effects

Tensile specimens of ATJ graphite used for support blocks, machined in the with-grain direction were coated with about 0.003" niobium carbide. Apparent loss of strength at room temperature was about 40% and at 1500 °C (2730 °F) about 15%. The strength reduction was found to be much less in compressive loading so that the effect of the coating poses no problems for the compressively loaded support blocks. A test on a carbide-coated H-205-85 ring-shaped specimen at 2704 °C (4900 °F) showed a 17% strength gain compared with a bare control specimen, but the coated specimen deflected 58% more under load.

Thermal expansion tests on hot-pressed NbC and TaC were initiated to define coefficient of thermal expansion of the protective coatings from room temperature to 2500 °C (4532 °F).

e. Hydrogen Corrosion

A quality control test was developed to assure the corrosion resistance of niobium carbide coatings on support blocks under conditions approximating the reactor operating environment. The tests are conducted in conjunction with fuel element tests in the element hydrogen corrosion apparatus. The sample is a hexagonal-shaped, one-seventh section of a coated support block. They are positioned at the exhaust end of the test fuel element in such a manner that all of the hydrogen from the element test passes through the support block section coolant holes. Since the block sections are not electrically heated, they are exposed to a temperature lower than that of the element. The nature of the test apparatus precluded direct measurement of the temperature of the

block or of the exit gas. Therefore, a modified thermal capsule device has been fabricated to determine test temperatures. The elements associated with the above tests operated at temperatures ranging from 2149 (3900 °F) to 2760 °C (5000 °F). Many of the block sections were exposed to two five-minute runs at temperature and some survived three five-minute runs. For the most part, the blocks behaved well, and exhibited no indications of coating corrosion or defects except when run with below-average fuel elements which affected behavior of the blocks.

Recent results from tests to evaluate the corrosion of uncoated graphite in hydrogen indicate that silica impregnation increases the corrosion rate of H4LM and ATL grades of graphite in the temperature range of 1500 to 1800 °C (2732 to 3272 °F). Low-temperature testing approximating service conditions ($\approx$ 420 °C (788 °F) and 700 psi) are not possible in the present experimental equipment. These data reveal that the corrosion rate at the temperatures of the test for impregnated H4LM is approximately a factor of 10 greater than that for virgin H4LM. For grade ATL, the corrosion rate ratio between impregnated and virgin material is approximately a factor of 4. It remains to be proven whether impregnated graphite corrodes at an accelerated rate at actual inner reflector operating temperatures and pressures.

f. Water Adsorption Effects

The effect of water at liquid nitrogen temperature on the flexural strength of graphite grades ATJ and H4LM has been studied to determine if adsorbed moisture might have harmful mechanical effects during reactor operation. Preliminary tests were conducted on both dehydrated and water-saturated samples at room temperature and at cryogenic temperature. A general increase in strength was noted and no harmful effects were observed, but further investigation is needed.

3. Cluster Plate Materials

A successful powder-metallurgy fabrication procedure has been developed for 304-type stainless steel containing 1.2% Gd_2O_3 for neutron absorbing cluster plates. Two small compacts (5-7 lbs) were rolled to .190" thick strip without edge cracking or surface

tearing, which had been a severe problem. Success in rolling the compacts was due to the presence of a roll lubricant, lower reductions and reduced roll speed. Using the same procedure, a 40 lb. compact (2" x 6" x 20") was rolled to strip on a rod mill. Due to the difficulty in handling a large compact, a few edge cracks occurred. Later, 40 lb. compacts were rolled with negligible edge cracking. The surface finish was again good. Mechanical property data representing the hot-rolled, annealed condition were quite consistent. Respectively, the values of ultimate strength, yield strength and percentage elongation were 81,500 psi; 29,300 psi and 69% at room temperature. The vendor attempting to develop melting, casting, and rolling procedures for gadolinium-containing stainless steel achieved success, for production quantities of three and five inch wide material have been received. This vacuum-melted material meets PD Specification 30057 in all respects except for an insignificant deviation from the manganese content. Both phases of this work have been reported. The evaluation of other cluster plate materials (silver-indium-cadmium, silver-indium-cadmium-tin, and 1% boron-10 containing stainless steel) was completed and reported.

4. Beryllium Components

The metallurgical aspects of the reuse of beryllium reflectors, including radiation effects on tensile properties and swelling, and chemical purity considerations, have been reported. It was concluded that beryllium is not damaged by the NRX-A environment, and on this basis reflectors are reusable. The various aspects of the reclamation and use of AEC-owned beryllium scrap have been considered and reported. It was concluded that this scrap could be reclaimed for NERVA applications.

Microcracks can be developed during the machining of beryllium parts, and this damage can result in lower strength and ductility than normally obtained. The mechanism of this phenomenon and its causes and remedies are well understood and reported in the literature. However, data are lacking for cryogenic and elevated temperatures. Consequently, a program has been planned to evaluate the effects of surface damage on commercially pure structural beryllium (NERVA quality) and high-purity, electrolytically-reduced metal. The vacuum hot pressing and extrusion processes are included.

The properties of hot pressed material used for NERVA components were reviewed but could not be meaningfully analyzed, primarily because the quantity of data was insufficient. Additional data are being collected on a continuing basis for inclusion in the analysis when this becomes possible. Also, a sample from the first extruded control drum is being evaluated to determine the degree of anisotropy of thermal expansion and tensile properties. In addition, the effects of density on notched and unnotched tensile properties are being determined on specimens of known density machined from a porous part. Results have shown excellent correlation between porosity, as revealed by fluorescent penetrant testing, and density.

Corrosion of machined beryllium parts appears to have been caused by the use of chlorinated, sulfonated lubricants during deep-hole drilling although cleaning and handling techniques also could have contributed. A detailed investigation is being made to determine remedial actions.

5. Titanium Alloy Components

A microstructural survey was completed on 5% Al-2-1/2% Sn titanium alloy core bolts which failed during vibration testing of ND 201. The threads were not produced by rolling as specified. From the lack of cold work about the thread contour and from short cracks protruding inward from the threaded surface on a few bolts, it was deduced that thread grinding resulted in thermal (grinding) cracks. Examination of several large cracks revealed a transgranular path without metal deformation implying that failure occurred by fatigue. Several failed bolts were analyzed for hydrogen content because of the possibility of pick-up and subsequent embrittlement during plating. In all cases the hydrogen content was well below the solubility limit, as verified by microstructural observation which did not reveal the embrittling hydride precipitate.

Mechanical property data on the titanium-5% aluminum 2-1/2% tin alloy (ELI) furnished by titanium support ring forging suppliers have been analyzed and compared with typical properties data. Properties of all acquired hardware have proven to be equivalent or better than typical and add justification to the choice of this material. In addition, room temperature and cryogenic temperature smooth and notched tensile and impact data have been

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generated from closed die and ring rolled titanium support forgings. Room and cryogenic temperature tensile as well as room temperature fatigue data is currently being gathered on titanium sheet material.

6. Aluminum Alloy Components

The selection of 2219 aluminum alloy for the core support barrel was discussed in a topical report in terms of the functions of the barrel, the barrel design, material properties requirements, candidate materials and manufacturing practices. Based on this review the superiority of aluminum was demonstrated and the 2219 alloy was shown to be the most suitable of its class. The use of a modified aging cycle produced barrels with acceptable dimensions and properties. Tooling and processing were modified to assure that the barrel did not become oversize when subjected to the normal aging cycle $[(36 \text{ hours at } 177^\circ\text{C (} 350^\circ\text{F)}]$.

Specimens were taken from a quadrant of a scrapped core support plate forging (2219-T852 aluminum) and tested in tension as a function of location and direction in the original forging. The results indicated considerable variation in properties along the radius with a 40% decrease in yield strength from the periphery to the center. The magnitude of difference between the center and edge was greater than expected and its significance is being investigated.

7. Nickel Alloy Components

A metallurgical evaluation of the NERVA NRX-A tie rod fabrication process was completed and a report has been issued. For regular tie rods of Inconel X-750 and Inconel 718, the fabrication process is satisfactory; however, additional development work would be required to eliminate minute folds at the interface between the tie rod (tube) and the plug insert in instrumented tie rods. Included in the evaluation were tensile properties, metallography, dimensional contraction during the aging heat treatment and oxidation (scaling) during heat treatment. On the basis of a statistical analysis of the tensile properties of production lots, the strengths of Inconel X-750, in applicable process and material specifications, were raised from 80,000 to 90,000 psi yield and from 145,000 to 160,000 psi ultimate. Because of its high

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strength and freedom from significant radiation damage described later, Inconel 718 has been chosen for the tie rods in A-5.

The evaluation of an Inconel X-750 nozzle end interface seal which failed during a component test revealed the prior existence of cracks which could not be detected by ordinary non-destructive methods. Failure was deduced to have been promoted by stress-corrosion associated with prolonged acid pickling and/or cracking during aging of the material under residual tensile stresses. Other nozzle interface seals from the same vendor have been examined, and although of considerably better quality than the failed one, all had some defects. A report of the failure analysis has been prepared. A visit to a new vendor indicated that the seals now being manufactured from Inconel 718 are being properly processed and that the reliability of these new parts will be high. The insensitivity of the 718 alloy to age-cracking and stress-corrosion also reinforces our confidence in the new parts. The process specification for these seals has been revised to incorporate additional, needed quality provisions.

The mechanical properties and coefficient of thermal expansion of Ni-SPAN-C 902, used in NERVA bolting applications, have been determined at cryogenic, ambient and elevated temperatures. The data developed were used in changing the specification (PDS 30044) requirements for this material. The data also show that the material is notch insensitive, the tensile properties decrease with increasing temperature from -196°C to 260°C (-320°F to 500°F) and there is no significant variation in the thermal expansion coefficient for different heat treatments.

An investigation of TD Nickel, a thoria dispersion-hardened alloy of nickel, was conducted to determine the feasibility of using it in NERVA reactors. Although the planned investigation was substantially reduced due to redirection of the program, information from other test facilities was assembled for evaluation. The conclusions, which have been reported, are encouraging enough to consider TD Nickel and a more recent, stronger modification for specific uses. Although not remarkably strong at normal temperatures, these alloys retain their strength much better than other alloys at temperatures approaching the melting point.

8. Seizing and Galling Studies

Because of the need to reassemble reactors and subassemblies and to reuse components, seizing and galling must be avoided. Therefore, an apparatus especially designed to evaluate the ability of various coatings to prevent seizing of NERVA fastener materials has been built. Initial tests showed the need for slight modification of the apparatus and specimen designs. Some materials have been received for testing including Ti-5Al-2.5Sn, Ni-SPAN-C 902, 303 Se and 304 stainless steels and A-286 high-temperature alloy.

C. Fabrication Development

1. Studies of Adhesives and Cements

RTV-891 Silicone adhesive has been tested for its ability to maintain a leak-tight seal between the stainless steel orifice jets and the channel wall of the fueled elements. Tests indicate the seal was maintained at -196°C (-320°F), after exposure to 282°C (540°F), and after irradiation at -196°C (-320°F) to an integrated dose of 10^{16} nvt ($E > 1$ Mev). This information has been reported.

Simulated graphite containers for thermal capsules have been cemented in slotted fuel element channels with P-514, a carbonaceous cement. Since the curing process for this cement in this application is limited by the melting point of aluminum (the lowest melting thermal capsule material) all P-514 test joints have been cured at 127°C (260°F), where polymerization of the phenolformaldehyde binder of P-514 is complete. Rapid heating of these test samples at 2500°C (4532°F) caused this cement to crack and, in general, destroyed the bond between the element channel and the capsule container. Because gas-born particles are undesirable during engine operation, the deterioration of this cemented joint at 2500°C (4532°F) is unacceptable. In order to eliminate the cracking, macerated graphite felt was added to P-514 to change its consistency to thick, viscous paste. Samples cemented with felt-modified P-514, cured at 127°C (260°F), did not crack or deteriorate after rapid heating to 2500°C (4532°F), at a rate of approximately $40^{\circ}\text{C}/\text{sec}$. This development has been implemented with specifications for the material and the process.

For the now terminated NRX-B study, both 7 and 19 element cemented clusters were prepared in 12" lengths with single-hole ORNL and 19-hole WANL elements. One 19 element cluster was prepared with stepped ATJ end blocks. All cluster joints, which were cemented with P-514 cement, were graphitized at 1900 °C (3452 °F). Thermal shock tests (heating in helium to 1900 °C (3452 °F) in three minutes) had no effect on the clusters, including the one with the ATJ end blocks. Because this program was cancelled, evaluation of these short-length clusters was only partially completed, but the accomplished work will be reported.

Work on a process for cementing a non-fueled end cap on the nozzle end of the fueled elements has been assisted by this project.

The shear strength of the carbonaceous joint between the filler block keys cemented to both fueled and non-fueled elements has been determined. The specified design load requirement was ten pounds, whereas the load determined by tests averaged 100 pounds on both types of elements. Cemented graphite samples have been tested to determine shear, tensile, and compressive strength of an epoxy cement and a carbonaceous cement. The former is stronger at -196 °C (-320 °F) than at room temperature, but the latter, cured at 127 °C (260 °F) loses strength at -196 °C (-320 °F). Also, the tensile strength of graphitized, carbonaceous cement joints have been determined at room temperature, 870 °C (1600 °F), 1900 °C (3452 °F), and 2500 °C (4532 °F) in helium. This work will be reported this year.

2. Silica Impregnation of H4LM Graphite

H4LM graphite is the only commercially available graphite that embodies the strength and size requirements of the inner reflector cylinder of the NERVA reactor. Since this grade of graphite is inherently porous and permeable to gases, an impregnation treatment is required to prevent undesirable hydrogen flow through the wall of the reflector in reactor operation. During the past year, an experimental program was conducted to explore and optimize the presently used silica impregnation process developed by LASL.

A vacuum pressure impregnation apparatus capable of operation at pressures to 150 psi was designed and constructed for processing experimental samples. In addition, equipment for measuring the permeability of graphites from 1/2" to 2" thickness at up to 200 psi differential pressure was obtained. Results of the study of the silica impregnation process have shown that decreased permeability of H4LM to hydrogen is obtained by impregnating at pressures on the order of 80 psi rather than at 15 psi as specified in the LASL process. It was conclusively demonstrated that machining impregnated graphites results in increases in permeability that are substantially greater than predicted by the dimensional change. As a consequence, these data were factored into the existing specification with the result that increased processing pressures are now required and machining after impregnation is not permitted.

Determinations of the moisture content of impregnated H4LM graphite indicate that it contains up to 2% moisture immediately after impregnation. The moisture content decreases with time, however, and approaches an equilibrium value of approximately 0.1% after only about one week exposure to ambient conditions. Impregnated graphite that was previously dried at 170 °C (340 °F) attains equilibrium at approximately 0.1 w/o moisture in about the same time period. Permeability measurements on RVA, CFW and H4LM impregnated with different processes and materials have revealed that H4LM impregnated per PS 294545, the process used for NERVA reflector cylinders, exhibits the lowest permeability measured to date.

3. Control Plates

The manufacturing procedures employed in producing control plates for NERVA reactors have been similar to those developed by LASL for making KIWI plates. Some changes have been made, however, to adopt the process to WANL equipment. The procedures have been reported, and the results of tensile tests, fatigue tests, and thermal expansion measurements of the Al-B¹⁰ control plates for the ND 103 and 104 was completed by the WANL production facility. Experience with the ND 103 plates showed that about 10% were lost because of edge cracking during rolling; in addition, one plate was cracked in forming, two did not meet

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specification because of difficulties in punching, and one had a minor deviation in dimension because of shearing trouble. Thus, about 84% of the potential product was available as deliverable hardware. Considerable experimentation was carried out with the object of minimizing scrap rate in production. The most recent experience shows that as a result of this effort, the loss of plates caused by edge cracking during rolling of the sheet has been essentially eliminated. The improvement cannot be ascribed to any one change in procedure; rather, it seems to reflect the cumulative effect of close attention to, and careful control of, a number of steps which are minor factors individually. A process has been evaluated and found satisfactory for the recovery of B^{10} from the scrap generated in production. Production plans are dependent upon this recovery.

4. Thermal Capsules

Calibration of thermal capsule wires of 27 compositions was accomplished by observing the change in shape of the wires upon melting. Melting temperatures ranged from 660 °C (1220 °F) to 2850 °C (5162 °F). Difficulty was encountered with most high-temperature Nb-Ta alloys, pure W and pure Ta wires. These materials form a carburized, higher melting-temperature surface during testing which at times does not deform even when melting has occurred in the interior of the wire. Therefore, simple observation of the wire tip shape is inadequate for these materials, and the detection of internal porosity by radiography or internal eutectic formation by metallography may be required. A report of this work has been made.

5. Metals Joining

The electron beam welding process for assembling end caps to control drum assemblies was finalized. Careful selection of amperage and the use of high (175 ipm) welding speeds eliminated the need for filler metal. The narrow bead that characterizes this high speed process reduced distortion to a point where the need to grind journals after welding (and thus generate dust near the finished assembly) has been virtually eliminated. The application of the process involved a redesign of the joint configuration. The process development and application have been reported.

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The final qualification of the high frequency induction brazing process for brazing the instrumentation leads to the seal block was accomplished and the process itself was applied to the NRX-A1 reactor. It was established that properly produced joints can withstand 1000 psig at -320°F . The method of drilling through the bi-metallic plug was found to be critical. The drilling operation smears non-wettable stainless steel on the copper portion of the joint. The process development work has been reported.

A modified instrumentation seal plug procedure was developed based on the availability of a lower frequency power supply and the requirement to eliminate all water cooling in the reactor assembly area. A set of transmission leads 75 feet long was designed to carry the induction power (4.5 kw) without resorting to water cooling. An air-cooled split coil was designed to heat the work piece. The deeper penetration of the low frequency permitted a redesign of the brazing alloy preform to improve reliability. The process is being qualified.

A number of special joining processes have been developed (or are under evaluation) for assembling instrumentation into the reactor. A special fluxless soldering technique using a 95Zn-5Al solder was developed to join stainless steel instrumentation to boron-aluminum control plates for 500°F maximum service. A high reliability brazing process for joining pressure lines without introducing flux into the system was developed. The process involves a special fitting that permits highly localized heating and chilling to dam the flow of flux into the joint. A localized resistance heating technique was adopted.

After the basic fabrication techniques had been developed for the reactor, much of the metal joining activity was directed toward the assembly of instrumentation. Capacitor discharge welding was evaluated for attaching light gauge aluminum strips to structural components and has been reported.

Controls have been established for the attachment of strain gauges to non-critically stressed structures by spot welding rather than by cementing. The spot welding process is being metallographically evaluated with regard to its potential effect on highly stressed parts.

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6. Metals Treating

The initial attempts to provide required mechanical properties for structural aluminum components and at the same time maintain the unusually rigid dimensional control of large cylindrical sections resulted in the evaluation of several special heat treatments. The first of these establish special cooling rates from the solution temperature thus assuring adequate response to subsequent aging treatments without losing the dimensional control needed for subsequent forming operations. The second program quantitatively determined the dimensional effect of a short-time aging treatment which minimized dimensional changes during aging and conferred more-than-adequate properties for initial reactor operation. This program also evaluated the dimensional change that occurs as a result of the more complex long-time aging treatment needed for advanced operational conditions. The data from the two procedures was applied to the manufacturing programs and the necessary controls to assure the reliability of the process were applied. This work is being prepared as a report.

The effect of thermal processing on instrumentation that is attached to structures during processing (for example, instrumentation passing through the seal block during brazing) was reviewed to determine what processes would be applied.

7. Metals Forming

A program was initiated to determine the effect of forming stresses on dimensional changes during aging. This information is being evaluated and will be included in the data on heat treatment of aluminum.

8. Metals Treating (Surface)

Initial plating systems associated with the seal block brazing process blistered when subjected to the long brazing periods associated with reactor assembly. A special high-adhesion, low-efficiency system of gold plating was developed to prevent blistering and assure uniform plating thickness. A special bath sequence prevents accidental reduction-plating which has a characteristically low adherence.

Attempts to utilize brush plating for applying the gold film failed. This manually controlled process results in uncontrolled current density and perhaps erratic reduction-plating conditions. These conditions caused blistering under extreme conditions encountered in brazing. All phases of this plating work have been reported.

The latter portion of the period was marked by the need to divert available funds and manpower from process development to process control as previously developed processes were applied by vendors. The process control activity reviews the building block processes (i.e., TIG welding, sizing of cylinders, heat treatment, etc.) in light of the specific industrial environment in which they are being applied. It is the aim of this new phase of activity to improve the manufacturing reliability of the basic processes involved in reactor construction while maintaining or improving the engineering reliability.

The initial effort involved a study of factors influencing weld soundness in 2219 aluminum welds. Cleanliness was the major factor and the specification was modified to highlight the aspect of process control. Radiographic controls were added to monitor adherence to the cleanliness requirements. This work will be reported later this year.

D. Irradiation Effects

1. Structural Materials

Four candidate tie rod materials, Inconel X-750, Inconel 718, Hastelloy R-235 and A-286 were irradiated at -320°F at the Ground Test Reactor (GTR) of General Dynamics/Fort Worth (GD/FW), Texas late in CY 1963. The post-irradiation testing and data analysis were completed this year and reported. The neutron dose was not as high as expected, being equivalent to only one to four minutes of NERVA operation at 75% of full power (maximum dose, 7.1×10^{16} nvt, $E > 1$ Mev). This is satisfactory for the A2 and A3 hot tests, but higher doses are needed for subsequent tests. Results indicated no serious impairment of mechanical properties at this dosage level for any of the materials tested. However, a consistent 20 - 30% decrease in the elongation of Inconel X-750 and 718 specimens at -320°F after irradiation and room temperature annealing indicates loss of ductility may be a signi-

ficant problem at higher dosage levels. Changes in the properties of R-235 and A-286 were not as well defined due to the lower neutron dose received (less than one-half the maximum). Also property changes were masked by the variation in properties of unirradiated control specimens of these materials.

Due to the unexpectedly low dose for this 150 hr., GTR test, the two 200 hr. tests at GTR planned for CY 1964 were changed to one 400 hour cryogenic test in August, 1964, with an expected maximum neutron dose in excess of 10^{18} nvt.

Utilizing a technique, developed at WANL, for transferring specimens from the irradiation dewar to the cryostat on a "hard" beam tensile tester, a large number of specimens may be tested at -320°F before warmup. With this procedure the problems associated with radiation effects on the NASA in-pile tensile testing apparatus can be avoided. Also, the number of specimens tested before warmup can be greatly increased. Materials selected for the 400 hr. irradiation were: Inconel X-750, Inconel 718, Ti-5Al-2.5Sn, spring temper AISI 302 stainless steel, spring temper AISI 301 stainless steel, beryllium (hot pressed), aluminum 2219-T852, aluminum 2219-T31, RTV 891 orifice cement, and H4LM graphite.

Tensile and notched tensile data are to be obtained for the metal samples in the following conditions:

- (1) At -196°C (-320°F) before warmup.
- (2) At -196°C (-320°F) after warmup to 27°C (80°F).
- (3) At 27°C (80°F).
- (4) At -196°C (-320°F) after one hour anneal at expected decay heating temperature.
- (5) At expected decay heating temperature.

A replication of three was chosen; for graphite the replication was 10 because of its usual variability. Graphite specimens are to be tensile tested at 27°C (80°F) and -196°C (-320°F) following warmup to 27°C (80°F). The cemented orifices will be leak tested and shear tested at 27°C (80°F). In addition electrical resistivity measurements may be made on Inconel X-750 and Inconel 718 in an effort to study damage mechanisms, if satisfactory apparatus can be developed. A few stainless steel

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helical springs will be irradiated under load to study radiation-induced stress relaxation in springs.

This single 400 hr. cryogenic test will subject most of the material selected for cryogenic irradiation to a dose equivalent to 10-20 minutes of NERVA operation and annealing treatments simulating decay heating temperatures. Additional testing of the materials selected for this test at higher dose levels is desirable from the standpoint of increased reliability and performance and may be required once the data have been analyzed.

2. ZTA Graphite

ZTA graphite irradiated a year ago at the GTR to 6×10^{16} nvt ($E > 0.1$ Mev) increased in tensile strength from 3850 psi to 4320 psi in the with-grain direction. The tensile strength in the across-the-grain direction showed an increase from 1174 to 1604 psi. This represents an increase of approximately 25% for both directions. Thermal expansion was not affected by the irradiation. A report describing these results has been prepared. The long delay in this work was due to the need for a design of tensile specimen to assure fracture in the reduced section.

3. Shield Materials

All work on the lithium hydride shield capsule irradiation program was completed and reported. It was found that at temperatures less than 130°C (266°F) and integrated fluxes up to 2.0×10^{16} n/cm² no swelling of the capsules occurred. At higher temperatures the extent of swelling increased with temperature. A maximum diametric expansion of 3.06% was observed at 3×10^{16} n/cm² and 250°C (482°F). The capsules containing sintered pellets swelled less than those containing non-sintered, cold-pressed pellets. There were no changes in capsule dimensions as a result of post-irradiation heating up to 300°C (572°F).

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4. Post-Operative Examination of NRX Reactor Materials

Plans for post-operative examination of the materials in ND 101, ND 10202, and ND 103 were prepared and incorporated in the comprehensive plans for the respective tests. Extensive modification of these plans was made because of budgetary changes and equipment limitations. As a result, more of the mechanical and physical tests will be performed by the Materials Department. For some components, this required holding samples until radioactivity had decayed to a level which would permit them to be handled with facilities available in this laboratory. Examination of the NRX-A1 materials was for the most part limited to dimensional checks, non-destructive testing, and visual inspection, because destructive tests of apparently undamaged components were not permitted. Because of the nature of the A1 test, this restriction is not particularly severe, and in subsequent tests of hot reactors, it will not apply.

E. Specifications

In response to the continuing need for specifications, approximately 26 new materials specifications and 22 new process specifications were written and issued by May, 1964. During this same period, approximately 39 material specifications and 31 process specifications were revised. Supplier and customer comments, experience with core hardware, materials development data, and changing design requirements formed the basis for these revisions. Bi-weekly listings of new specifications and semi-annual compilations of all WANL specifications were issued.

All material specifications have been submitted to suppliers for review and new specifications are submitted as they are written. A program to discuss specifications with these suppliers has been initiated.

The responsibility for equipment specifications was assigned to the Materials Department and principal effort has been toward accumulating the data on existing specifications. Six WANL equipment specifications have been written and seven have been revised during this period.

Constant surveillance has been maintained over Federal, Mil and commercial specifications to insure up-to-date files of these documents.

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Particularly noteworthy were the specifications requiring major effort. The single group of specifications requiring the greatest effort was that for the manufacture, coating, inspection, and packaging of fuel. Others included pyrolytic graphite specifications which now have in-process, chemical, macrostructural and microstructural standards; vacuum hot pressed and extruded beryllium specifications which now more closely reflect the capabilities of the beryllium industry, and which for the first time in this industry, have ultrasonic flaw detection requirements; materials and processes specifications for several adhesives and cements; titanium alloy forging specifications; tie rod material and process specifications; and cleaning and clean room specifications.

Design review and drawing sign-off to uncover potential problems and approve the choice of materials and processes continued to require a significant effort.

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PROPOSED CY 1965 REACTOR NON-FUEL MATERIALS PROGRAM

A. Vendor Liaison, Vendor Evaluation and Consulting

Vendor liaison, vendor evaluation and consulting will continue to require significant effort to improve the NERVA product and the delivery and, wherever possible, the cost. Although vendors of most materials used in NERVA will be contacted, emphasis still will be on the graphite vendors. This is due to the combination of our critical technical requirements for graphite, both conventional and pyrolytic, and the lack of standardization of products by the graphite-producing industry. The objects of these efforts are:

1. With all pyrolytic graphite manufacturers:
 - a) To continue evaluating manufacturers for progress in solving present problems
 - b) To improve specification as vendors' progress permits
 - c) To help develop better products
 - d) To examine new products which may do existing jobs better
2. With conventional graphite manufacturers:
 - a) To continue the evaluation of their processes, facilities and grades to solve a number of existing problems
 - b) To improve specifications as vendors' progress permits
 - c) To help develop better processes and/or grades and/or larger sizes of existing grades to solve such problems as exist in obtaining consistently higher quality inner reflectors.
 - d) To evaluate new grades and/or products which may do existing jobs better

Consulting will continue to be a major effort, for numerous requests for advise on materials come from design, manufacturing, quality control, purchasing and suppliers' personnel. Typically, help is needed on selection of materials, the properties of materials in selected environments, fabricability of materials, methods of evaluation of materials, determination of those properties needed in specifications, etc. An earlier section of this report outlined specific problems encountered in CY 1964. Similar ones are expected in

CY 1965, but their day-to-day nature precludes advance planning. However, this is one of the most important aspects of the Materials Program.

This phase of the process development activity is expected to continued to require a major effort for CY 1965. The daily problems of reactor assembly, i.e., last minute instrumentation difficulties, electrodeposition, anodizing, brazing, and a host of other things have historically required considerable effort. Consultation effort on design, producibility, aiding in the evaluation of a new vendor, helping the vendor to develop a controlled process and other problems in the welding, brazing, and fabrication of components also are expected to demand instant attention. Technical requirements for process specifications must also be developed as part of this work.

B. Materials Development, Evaluation, Application and Behavior

The continued advances in reactor design are becoming increasingly dependent upon the capability of materials. Proper support of the current manufacturing programs by materials efforts as well as by more advanced work in anticipation of future requirements is indicated. The more significant work areas in the realm of materials studies are: continued evaluation and tabulation of mechanical and physical property data, extension of design reliability through selective testing and statistical evaluation, study of failure mechanisms of materials of potential interest, and development of new materials when necessary.

1. Materials Data Sheets

Work will continue in the preparation of the remaining data sheets for inclusion in the NERVA Materials Properties Data Book. Also, revision of the component-materials-properties requirements list will be completed and new or changed requirements for materials properties data will be entered on the worksheets. The revised list will dictate the direction of efforts spent on data compilation. Specific properties for many materials are still needed to assure reliable designs and justifications for particular selections. The increasing number of requests from other departments for properties data also requires a more systematic approach to simplify data retrieval and subsequently enter this information on data sheets.

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For example, statistically significant physical and mechanical property data in the form of Materials Data Sheets are required for pyrolytic graphite as functions of crystallographic orientation, microstructure, testing temperature, and testing environment to allow efficient design of reactor components. Standard test specimen designs and test techniques for physical and mechanical properties will be established. Such properties as thermal and electrical conductivity, tensile strength, flexural strength, fatigue strength, Young's modulus, coefficient of thermal expansion, Poisson's ratio, creep and thermal shock resistance will be determined as functions of the above variables. Materials Data Sheets will be generated as data become available.

2. Graphite Studies

Significant advancements were made towards understanding of NERVA engine applications and requirements and in some cases problems arose which demonstrated the need for more detailed or expanded work in certain areas. These include:

a) Improved Inner Reflector

The evaluation of materials for inner reflector application confirmed the relatively low and variable quality and strength of graphite available in the required size. The necessity for either improved graphite in large sizes or component redesign to utilize the superior grades of graphite available in smaller sizes was established. Earlier attempts to improve the properties of the inner reflector cylinder included a proposal to SNPO for the scale up of RVA grade graphite to the length required for NRX-A reactors. This was not successful at the time due to financial, not technical, limitations. The successful performance of the inner reflector cylinder is vital to the reactor performance and continued emphasis on material and design improvement is a necessity.

Full size pieces of several graphite grades will be obtained for potential use in a segmented-type inner reflector cylinder. The grades being considered have been evaluated in smaller sizes and were noted to have substantially higher strengths than presently available in H4LM grade graphite. This program will be divided into two main tasks which will run concurrently -- graphite properties determinations of full size shapes and cementing development

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of sections to form seals for segmented cylinders. An evaluation of the mechanical and physical properties of full size graphite sections will be made as a function of orientation and temperature. Full size segments for this evaluation will include rings stacked to produce a full-length cylinder and full-length staves. Procurement of sufficient pieces to permit construction of a full-size segmented cylinder will be based on the materials evaluations and the requirements of design. This aspect of the program will be coordinated with and support the full size component test program of the Mechanical Design Group. Preliminary estimates indicate that the cost of this work may fall within the capabilities of this project. If not, these large scale pieces will not be obtained.

A study will be made of cements which can be used for the graphites being considered. The short range objective is to provide a cement which will promote a sound bond between the graphite segments and thus impede gas flow through the segment joints. The long range objective is to provide a mechanical joint between segments which will resist deterioration due to the thermal and neutron environment during operation of the reactor and perform a structural function in the design.

b) Evaluation of Promising New Structural Graphites

The demand for increased reliability in present reactor designs and more severe environmental conditions for longer operating times has necessitated a continuing evaluation of newer grades of graphite to meet these needs as well as the generation of more detailed data on currently used grades. This work will be continued and coordinated as closely as possible to reactor design requirements. The strongest structural graphite available during the CY 1964 was an isotatically molded grade, known as PO-3, made on an experimental basis by one of the smaller manufacturers. Initial evaluation revealed exceptionally high strength as follows: Tensile strength, 5,500 psi; flexural strength, 8,000 psi; and compressive strength, 20,000 psi. This material will be investigated further to define its availability and the design properties. The use of the isostatic molding process possibly will permit pre-forming of many components such as support blocks, seal segments, etc.

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c) Annealing of Pyrolytic Graphite

It is presently known that annealing of pyrolytic graphite causes large dimensional changes of up to 16% in the across-grain direction and up to 6% in the with-grain direction with associated changes in physical and mechanical properties. However, these changes are not known specifically as functions of the degree of annealing, type of microstructure, annealing environment, or of applied compressive or tensile loads. Such information is required for designing components and systems and for defining the optimum condition of the material for each component. These changes in pyrolytic graphite caused by annealing will be determined as functions of time-temperature cycles, type of microstructure, environment, and applied load during annealing. Data on the effects of annealing will be generated on each engineering property. Annealing temperatures will range from 2200 to 2400°C (3992 to 4352°F) and times from 10 minutes to 10 hours.

d) Alternate Pyrolytic Graphite Materials

Problems associated with the procurement and quality of vapor-phase-deposited pyrolytic graphite components indicated the need for other approaches to both the production and subsequent evaluation of pyrolytic components. Studies were initiated and are continuing to develop composite pyrolytic components and to develop non-destructive evaluation techniques for these components, particularly the measurement of orientation effect on thermal conductivity.

Presently all PG components are machined from as-deposited material. Because of warp and bow in the material, a component is machined by cutting across many basal planes, thus increasing the effective across-grain thermal conductivity. Residual stresses cause failure of many components during machining, inspection, handling, etc. Furthermore, deposition defects (i.e., nodules, sooting, etc.) reduce manufacturing yield and operating reliability.

Insulating sleeves and tiles made from wrapped and laminated pyrofoils will continue to be evaluated for mechanical properties, temperature stability (including thermal shock), thermal conductivity, hydrogen corrosion resistance and manufacturing reproducibility. Such materials as glassy carbon, pressed pyrofoam, and pyrolytic boron nitride will be reviewed and evaluated as possible materials for the manufacture of insulating cups. Where required, forming methods, bonding materials and procedures will be developed. Promising processes

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such as deposition to final size and strain annealing to reduce stresses will also be evaluated. Evaluation of mechanical properties, temperature stability, and thermal conductivity of Grafoil-100 tape and Grafoil-101 laminates will also be conducted. A similar evaluation of Graph Tape will be started immediately upon receipt of material.

e) Hydrogen Corrosion of Uncoated Graphite

Detailed analysis of the corrosion requirements of the uncoated lateral support, inner reflector, seal segment and filler strip components indicated the need for detailed information on the corrosion behavior of the various grades of graphite used as a function of pressure, mass flow, channel area, temperature, etc. This is particularly important considering the longer operating times and higher operating pressures and temperatures of future reactors. It is known that at high temperatures graphite is attacked by hydrogen; however, data that define rates of attack as a function of temperatures and pressures in the range of NERVA interests are, for the most part, not available nor can they be obtained from extrapolations of data generated under widely varying sets of conditions. To satisfy the need for this information, the following experimental program is planned:

A small scale experimental high-temperature, hydrogen-corrosion furnace capable of operation at temperatures to 3000°C (5432°F) and pressures to 1400 psi will be constructed. Corrosion rates will be established as a function of temperature and pressure for various grades of commercial graphite, graphites used in the NERVA lateral support system, pyrolytic graphites, and other graphites considered for reactor components in the future to provide corrosion rate input to the design groups and to possibly establish a correlation between inherent graphite properties and corrosion behavior. Geometry effects such as typical design gaps and slots will be evaluated for their effect on corrosion rate as well as the influence of gas flow rates. The corrosion behavior of NbC coatings deposited on graphites will be pursued to supplement the support block program by relating coating properties with corrosion resistance. The area of inhibiting corrosion by additions to the hydrogen stream and/or to the graphite will be explored. Also the subsequent

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deposition of corrosion products from the hydrogen gas will be examined. Dimensional and weight change will be the primary methods of evaluating experimental samples. These methods will be supplemented by porosimetry, resistivity, and other techniques as the need arises. Special attention will be devoted to developing an understanding of the mode of failure of the coating so the substrate coating interactions may be better controlled to provide longer life.

f) Hydrogen Corrosion of NbC-Coated Support Blocks

The function of the support blocks in the NERVA reactor is critical. The present design requires each support block to support at least six fueled elements under conditions of extreme temperature and high hydrogen flow. The success with which these components perform in reactor operation, is solely dependent upon the quality of the niobium carbide protective coating. This program is designed to measure the ability of the coating on support blocks to perform satisfactorily in the design environment. Corrosion tests with sections of coated support blocks will be run to define the effects of coating thickness, coating adherency and various sized defects on the ability of the blocks to survive in flowing hydrogen at high temperatures. In addition, the upper limits of operating temperature and times will be determined. These tests will be conducted in conjunction with fuel element corrosion tests and will be performed in the Fluid Flow hydrogen corrosion test facility. Frequent use will be made of beta-back-scatter coating thickness determinations and metallographic observations to correlate results. Information derived from this study will be fed back and factored into coating processes and quality control. Considerable effort will be directed toward assuring the quality of production reactor components. This effort will involve interpretation of hydrogen corrosion test results on process samples.

The coatability and quality of the coating of other grades of graphites being considered for support blocks for future reactors will be established. Metallographic evaluation, adherence tests, and corrosion resistance will be the primary tools of evaluation.

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g) Permeability and Impregnation of Inner Reflector Cylinder Materials

As a result of a program conducted during the past year it was shown that the permeability of the H4LM graphite currently used for the inner reflector cylinder could be substantially reduced by pressure impregnating with a colloidal suspension of SiO_2 in water. The process is considered to be optimized and no further work with H4LM is planned, since the permeability of the impregnated material satisfies design requirements. At present, several designs and grades of graphite are being considered for use in future NERVA reactor systems. First, measurements of the permeability of these grades of graphite indicate that in all cases the permeability is less than the permeability of H4LM but greater than the permeability of SiO_2 -impregnated H4LM.

The scope of work in this areas then is dependent on reactor design requirements. If the permeability of the as-produced graphites satisfies design criteria only permeability determinations will be performed. If the materials do not meet design criteria, a program will be conducted to determine first the applicability of the SiO_2 impregnation process to the raw materials and beyond that to develop more appropriate processes which do not require the use of SiO_2 . The presently available vacuum-pressure impregnation apparatus will be used for this work. Evaluation will be via permeability determinations on the as-produced and impregnated materials. In addition, the effects of the impregnant on such graphite properties as strength, coefficient of friction, moisture content, and corrosion resistance will be defined.

3. Beryllium Components

a) Mechanical and Physical Properties

Because the mechanical and physical properties data obtained on hardware procured to date have not been sufficient for statistical analysis, this program will be continued on samples of beryllium being procured for advanced reactors. It is expected that extruded material will be available for evaluation for the first time during this period. Emphasis will be placed on obtaining statistically meaningful data for hot-pressed and extruded materials from room to liquid hydrogen temperatures. Properties to be determined are notched and unnotched tensile strength, density, specific heat, thermal conductivity, and thermal expansion.

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b) Thermal Shock Behavior

The thin web of the outer reflector sector experiences a severe thermal gradient at start-up due to the flow of cold hydrogen. Because of the adjacent thick sections of the sector, the web is constrained so that cracking of the web is a possibility. Calculations show that extremely high stresses will be encountered, based on elastic stress-strain theory. In another program, thermal shock fractures have been found in specimens designed to constrain the zone exposed to a large thermal gradient. It is planned to assess the present knowledge and determine if further investigation is warranted. If so, a literature survey will be conducted to determine the best methods for evaluating the thermal shock behavior of beryllium under NERVA operating conditions, and appropriate tests will be conducted as required to support the Engineering Component Test program.

c) Aging of Beryllium

Several investigators have shown that the strength and ductility of quenched beryllium can be improved by reheating to moderate temperatures, and the improvements in properties have been attributed to precipitation phenomena. Iron, an impurity in commercially pure beryllium, was found to segregate to grain boundaries during aging. Previous work was conducted on material which had been rapidly quenched in the laboratory. Rapid quenching may be impractical for NERVA components, but significant quantities of the precipitating phases may be retained in solution in beryllium which is not annealed after manufacture; this is particularly true for extrusions, which are air cooled from the extrusion temperature. Therefore, coupons of extruded metal will be aged for various times in the temperature range of 700-1300°F, tensile tested, and metallographically examined; this will be done using commercially pure beryllium as well as extrusions manufactured from electrolytically-reduced metal. Thermal treatments, involving air cooling from moderate temperatures and aging, will also be conducted on commercially pure and electrolytically-reduced beryllium manufactured entirely by powder metallurgy techniques. If successful, the program would result in improved ductility in beryllium; in turn, this improvement would increase the resistance of beryllium to thermal shock.

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4. Hydrogen Environmental Effects on Titanium Alloys

The work to date has indicated that the hydrogen environment is not expected to have an adverse, embrittling effect on the dome end support ring in early NERVA reactor tests. This conclusion was based on calculations and experimental results on commercially pure titanium which measurably begins to absorb hydrogen at about 300°C (572°F). However, experimental work should be conducted to confirm this conclusion and to define the operating limitations of titanium alloy components in future longer operating systems. Also, in present reactor designs, the titanium alloy core band and some of the titanium bolts are exposed to steady state temperatures of 227-282°C (440-540°F) which are considerably higher than the 5°C (40°F) temperature of the dome end support ring, which make them susceptible to hydrogen embrittlement. Maximum conditions of hydrogen gas pressure, temperature, stress and irradiation dose should be established and used to determine if the conditions induce hydrogen pick-up by titanium. However, because it may be impractical to immediately include pressure, stress and radiation as condition variables, initial work will be done to establish the effect of temperature and hydrogen at near-atmospheric pressure. In addition to the above-mentioned work, the maximum hydrogen tolerance limit for the titanium alloy used in NERVA will be established. This can be done by observing the tensile properties as a function of hydrogen content. Currently, a Sieverts apparatus is being constructed for the purpose of introducing known amounts of hydrogen into test specimens.

5. Nickel-Base Alloy Components

a) Inconel-Type Alloys

Because of its superior tensile properties, Inconel 718 alloy has recently been selected for tie rods and nozzle interface seals. A program is needed to determine the effects of cold work, prior to aging, on the properties of Inconel 718 and X-750 to improve the understanding of the property changes which result from such procedures. For example, the nozzle interface seal is cold worked in critical areas prior to final heat treatment. Furthermore, manufacturing economies and design advantages may be realized from property improvements which may result after thread-rolling and direct aging of tie rods. The program will be conducted on sheet stock of the 718 and X-750 alloys which will be cold rolled or elongated in tension by predetermined

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amounts prior to aging. The samples will be evaluated after aging by means of notched and unnotched tensile tests, hardness tests and metallographic observations.

During reactor operation, the tie rods may be subjected to repeated cycling to relatively high stresses. Fatigue data for such stresses and for the operating temperature range of these tie rods are non-existent; therefore, direct-stress (tension-tension) fatigue tests of Inconel X-750 and 718 will be implemented to fill these gaps in the data.

b) Dispersion-Hardened Alloys

The high-temperature requirements of the reactor may limit the usefulness of some of the more marginal materials in the core such as the Inconel X-750 or 718 tie rod. Therefore, the thoria dispersion-hardened nickel alloy, TD Nickel, was examined in CY 1964 because of its high-temperature capability. Results indicated that its strength is superior to those of the super alloys at temperatures above 1038°C (1900°F), but the room-temperature strength is much lower. However, DuPont has developed a modification of TD Nickel, known as TDM, which includes a molybdenum addition for solid-solution strengthening. This gives the alloy a room-temperature yield strength of 167,000 psi and much improved high-temperature ductility, making it competitive with the super alloys. TDM apparently has all the advantages of TD Nickel -- namely, notch insensitivity at all temperatures, high thermal conductivity, and excellent stability and strength after long-time exposures at elevated temperatures. Preliminary results of stress rupture and fatigue data appear as good or better than those of other nickel-base alloys. The capabilities of these thoria-dispersed alloys warrant a thorough evaluation of TD Nickel and particularly TDM to provide substantial bases for selection of the materials. Some development effort may then be desirable. Based on the separate behavior of nickel alloys and thoria in irradiation environments, it was concluded that TD and TDM Nickel would not suffer excessive damage under NERVA operating conditions, nor would excessive induced radioactivity occur. Recent published data, however, suggested caution, for TD Nickel irradiated at 5×10^{20} nvt ($E > 1.0$ mev) showed nil ductility in tensile tests. Although this is a 100 fold greater irradiation than a 20-minute, full power NERVA cycle, it is not known how lower

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irradiation doses will affect this material. Emphasis is to be placed upon obtaining materials suitable for the ultimate 60 minute, full power requirement, as radiation damage may be severely limiting in this region. Complete cognizance of development programs by DuPont will be maintained, especially in the area of welding and brazing to other nickel-base alloys. When TDM material becomes available, a preliminary evaluation will be made.

6. Seizing and Galling of Components

Prevention of seizing and galling of NERVA fasteners is necessary to permit disassembly and reassembly of components without replacement of components. The methods for preventing seizing of NERVA fasteners are currently numerous. Examples are given by the widespread use of silver plating, Tufftriding, anodizing and dry film lubrication. It would be desirable to quantitatively compare them to establish one or two lubricating methods which could be used on the majority of NERVA fastener materials. More important than this, however, is the need to definitely establish material problem areas by testing combinations over various conditions of fastener load application. The equipment needed to accomplish these tests has been received and is currently being modified. It will give instantaneous readout of bolt torque and bolt load with an X-Y recorder coupled to both torque and tension load cells. Any combination of materials for the bolt and nut components can be tested. From the torque-load data it is believed possible to determine the onset of "sticking" or seizing and thus to rate the various combinations of materials as well as surface coatings. Although much of the knowledge of seizing and galling has been obtained by empirical methods, an attempt will be made to use known principles to minimize the amount of testing required. Such a program should yield data on the susceptibility to seizing of the various combinations of materials, surface treatments and lubricants of interest in the NERVA reactor.

7. Thermal Capsules

Considerable difficulty has been encountered with the calibration of the thermal capsule wires which span the temperature range 2400 to 2850°C (4352 - 5162°F). These wires, Nb-Ta alloys, pure W and pure Ta form higher-melting-temperature carbide surfaces during

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calibration as the result of a reaction between the metal wire and the graphite capsule. Therefore, they do not readily deform, making it difficult to detect melting by observing the change in shape of the wire tip. Internal melting and occasional porosity occur when these wires are heated above the true melting temperatures of the alloys during calibration, but this can only be detected metallographically (with the possible exception of gross porosity which may be detected radiographically). Metallographic examination after reactor runs will entail costly hot cell work. Therefore, substitute materials should be investigated. Possible substitutes include properly chosen oxide compositions. For example, ZrO_2 combined with another oxide(s) may provide systems which show melting in the above mentioned temperature range and freedom from reaction with graphite.

8. Instrumentation Materials Problems

Instrumentation devices are generally procured and used as complete entities for the purpose of sensing some change and giving an electrical signal which designates the type of change. This approach is quite successful for normal use of established instrumentation devices. The materials of construction for instrumentation devices are commonly chosen because of specific behavior such as electrical, magnetic, strain sensitive properties, coefficients of thermal expansion, etc., for the purpose of producing electrical impulses which are proportional to pressure, motion, temperature, or acceleration forces. The NERVA operating conditions, however, are extreme: for example, low and high temperatures, hydrogen environments, contact with carbon, and high-flux irradiation fields. The problems of vibration and attachment (without damaging the substrate structural part) to a wide variety of materials of construction none of which are common in ordinary structures are also encountered. Since the materials of construction in instrumentation sensors are selected for their particular electrical or other properties, these materials are frequently not suitable for environments in contact with carbon, hydrogen, cryogenic temperatures or elevated temperatures when simultaneously exposed to irradiation.

It has been demonstrated that some of the commercial devices have failed in cryogenic temperature irradiation environments and in high-temperature environments when

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in contact with carbon. Even when the instrumentation device is satisfactory the attachment method has occasionally not been able to withstand the environmental change or the method of use and attachment may damage the structural parts. A number of problem areas have come to attention largely by failures in an empirical environmental test although some occurred in reactor tests. Some problems have been solved by a change in materials or design. Part of these changes have been proven by retesting but much work remains to be done. Full scale reactor tests are used to demonstrate the feasibility of a design, to establish the satisfactory characteristic of certain materials of construction, and to collect operating and environmental data which will enable design improvements to be made. Since these reactor tests are extremely expensive, it is essential that the instrumentation devices have high reliability to insure the acquisition of data, and failures, therefore, must be eliminated. Problems which arise as a result of failures are costly because of lost data and time. Furthermore, this is too late because tight hardware schedules will not permit time to do the required development before making the necessary correction. This frequently results in alterations that are based on "guesstimates" or the undesirable alternative of delaying an assembly schedule or sacrificing the data by not using that type of instrumentation device.

Several methods are available to avoid or to correct the cause of failures. Occasionally another material has adequate electrical, magnetic or thermal properties to perform the same function, but will have sufficiently different mechanical properties to withstand the NERVA environment. In this event a substitution may be made. In the event that no other material has adequate properties to perform the same function, it will be necessary to change the design by using the material differently, i.e., supporting it in another manner or protecting it with a coating. Occasionally this action creates additional problems. For example, for high-temperature thermocouples, protection of molybdenum from eutectic melting in the presence of carbon required the coating of molybdenum components with tantalum. Since the tantalum would also be in contact with hydrogen, it was necessary to add an additional coating of tungsten or refractory carbides to prevent hydriding damage.

To correct this unsatisfactory train of events in discovering and solving problems, a detailed review of instrumentation devices, materials used, design, function, and environment

will be conducted. This review must consider the effect of temperature, environment, and irradiation on the materials of construction. The method of joining or attaching the device to the substrate material must also be examined. It will be necessary to review drawings of each device, parts lists, E specs, the vendor's fabrication method, the application in reactor mechanical design and the precise joining materials and methods anticipated. To be effective, this review must be done long before the scheduled time of procurement. Corrective action, if required, will then be possible. The expected types of correction include: materials change, design change, fabrication development, joining or attachment development, and improved inspection behavior.

Currently recognized problem areas which will receive attention include:

- a) Tungsten/tungsten-rhenium thermocouples
 - 1. Development of brazing technique for sealing tantalum to stainless steel.
 - 2. Development of improved coatings for tantalum sheaths or alternate means for hydrogen protection.
 - 3. General problems in selection of other thermocouple materials for insulation, etc.
- b) Transducers - Pressure and LVDT's
 - 1. Optimize materials selection.
 - 2. Improve electrical insulation.
 - 3. Conduct post-mortem examinations.
- c) Strain Gages
 - 1. Evaluation of attachment materials.
- d) Test Fixtures
 - 1. Corrosion resistance during irradiation in water, as in PBRF irradiation tests.
 - 2. Metal joining techniques.

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9. Other Materials Problems

In addition to the above materials problems, there are others that require substantial efforts to determine the feasibility of using alternate materials for specific components. One particular case is that of the lateral support spring material, full hard 301 stainless steel, which has shown indications of fretting in simulated tests and does not appear strong enough for NRX-A5 design requirements. The Cu-Be alloys or modifications of the austenitic stainless steels, such as Zerolled 301, are possible alternate selections. Consideration of these substitutes requires mechanical testing to determine their suitability with regard to design parameters.

Another problem area involves the titanium bolt that couples the aluminum core support plate and the titanium core support ring. The aluminum plate has a thermal expansion coefficient much greater than that of the titanium bolt, which causes difficulties in maintaining dimensional compatibility during temperature variations. As a result, a corrosion resistant steel known as AMS 5624 looks desirable as an alternate material since it has expansion properties similar to those of aluminum at room temperature. To qualify the material for use, considerable mechanical and physical property testing is needed as a function of temperature. Due to the limited availability of material and lack of property data, this evaluation would have to be quite detailed. Anticipation of possible hydrogen embrittlement conditions in the titanium core band for longer operation at higher temperatures, has stimulated requests to evaluate the low-expansion alloys of Fe-Ni as a substitute material. Such a choice would provide a lower expansion similar to that of the graphite. The investigation will include the determination of the properties for conventional alloys of the Invar type as a function of temperature.

Other items, such as BeO and other unusual oxide properties for instrumentation design will also require definite efforts as will the search for radiation and corrosion resistant brazing alloys. More complete chemical impurity categorization is required of many materials for use in nuclear analysis. Other materials problems, unforeseen at this time, are certain to require attention in CY 1965.

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C. Fabrication Development

1. Joining of Graphite to Itself and to Other Materials

The application of cementing as a graphite joining technique received considerable attention in CY 1964. It will be expanded to include such problems as joining techniques for sealing purposes as well as strength for the segmented approach to inner reflector fabrication. As design requirements and lifetime of graphite hardware in core environments become more stringent, those joining and potting materials and processes which have been satisfactory for NRX-A2 may not be sufficient. A program will be initiated to determine the effects of irradiation and hydrogen corrosion upon integrity and the mechanical strength of these cements.

a) Carbonaceous Cements

The carbonaceous cements have been used in the A2 core as a means of joining filler block keys and thermal capsule containers to fuel elements. It is essential to determine the combined effect of temperature and irradiation on these joints. Since the thermal capsule cement has a filler composed of chopped graphite felt, it is expected this joining material would have a greater resistance to irradiation; however, this can be determined only by tests.

Other cement formulations will be developed to decrease the inherent porosity of carbonaceous cements after carbonization or graphitization. Permeability measurements will be used to determine the degree of impermeability of each cement formulation considered, since metallographic examination of cemented joints is unsatisfactory for this purpose. Special attention will be directed to determination of the effect of evolved reaction gases upon components when these cements have not been pre-treated at temperatures above the reactor operating temperature. In addition, more effort will be devoted to joining pyrolytic graphite with carbonaceous cements. Because of its freedom from pores, pyrolytic graphite is difficult to bond. This effort will be aimed at developing the necessary technology.

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b) High-Temperature Organic Structural Cements

Recent work in polymer chemistry has given rise to new cement formulations which can be used at 1000°F (538°C). These cements are composed of polymeric chains whose aromatic nuclei are joined by non-aliphatic bonds; therefore, this type of cement may be more resistant to irradiation, since aromatic polymers are generally more stable than aliphatic polymers. These adhesives can be used in a variety of applications if they have sufficient resistance to irradiation. Some examples of applications, which will be investigated, are (1) joining of instrumentation to the graphite inner reflector; (2) repair of the inner reflector; and, (3) sealing and joining of graphite segments for fabrication of a multi-segmented inner reflector barrel for future core design.

c) Ceramic Adhesives

Because of their high-temperature capabilities, inorganic adhesives were considered for NERVA application. However, most commercial ceramic adhesives are unattractive for bonding graphite because (1) the glassy-type adhesives must be melted (or matured) at elevated temperatures and must contain carbide-forming compounds in order to wet graphite; (2) they are inherently brittle; and, (3) they are susceptible to thermal shock unless the adhesive layer is extremely thin. Also, those ceramic adhesives which contain water in the "as-cured" state fail by the loss of water of hydration. At this time, the development of ceramic adhesives has not advanced sufficiently to warrant investigation for bonding graphite.

2. Control Drum Plate Assembly Fabrication

Control plate assemblies for the NERVA reactors are being produced by the WANL Materials Department. Development of the fabrication process has proceeded satisfactorily, and there are no serious problems at this time. However, it is possible that additional work may be required to meet changing requirements in the normal course of development. Such work will be undertaken as the need is foreseen.

3. Process Development

Available funds and manpower will be directed toward process control studies. Study of two processes that are of paramount importance in the fabrication of the close-tolerance, predominantly cylindrical hardware items that characterize a reactor is planned. Other studies may be initiated on the basis of production or service experience.

a) Electron Beam Welding

This process is particularly attractive because it permits close control of dimensions. Experience with the process on aluminum indicates a materials-process relationship between such important items as beam penetration and weld metal cracking. Careful control of process parameters and effective repair procedures have minimized these problems to date. However, a better understanding basis of these relationships might improve the manufacturing reliability of the present application and also permit a profitable and confident broadening of the electron beam application.

This program would result in the definition of a series of qualitative materials-process responses which in turn would be applied to control alloy content, process parameters, tooling or joint preparation to assure a minimum of manufacturing processes.

b) Control of Dimensions in Formed and Heat Treated Metal Structures

The fabrication of large diameter close tolerance cylinders and rings by the roll and weld technique characterizes many reactor structural components. After initial fabrication these parts must be brought to size and subjected to heat treatment. Sizing operations include rolling, stretch forming and spinning singly and in combination. Heat treatments include miscellaneous stress relief followed by aging. Materials include aluminum alloys (6061 and 2219) and nickel-base alloys (Inconel 718, Inconel X-750). The results of this program would provide a series of allowances for dimensional change against which tooling could be checked. Further, preferred sequences of operations would be suggested to provide optimum control for parts that must maintain dimensional stability during service.

c) Process Development

It is difficult to predict the specific problems as many will be related to the design detail as it comes out of reactor design work in the future. However, effort will be directed toward simplifying the instrumentation seal brazing process to eliminate inlay brazing and gold plating. Other work can develop from the redesign of the inner reflector cylinder to an aluminum structure, particularly if new alloys are required for strength and if brazements are needed for cooling purposes. Core diameter increases may lead to the introduction of new problems associated only with size redesign changes.

The need for process development in the area of instrumentation devices for the cores and assemblies for irradiation experiments had just begun to be recognized during the NRX-A2 assembly era. The process work would complement the materials work also being done under this project. Included are soldering, brazing, and welding of components, such as the dissimilar parts of high-temperature thermocouples (Ta to stainless steel, Mo to stainless steel, etc.) establishing the effects of these processes on the base materials; investigation of methods for attaching thermocouples, strain gages and other instrumentation devices to various core components and the effects of such processes upon the materials; and, assisting with irradiation damage analysis and providing any special protection required for the devices during irradiation or to prevent undesirable interactions between the devices and the irradiation environment leading to contamination of the environment.

4. Miscellaneous Fabrication Problems

A wide variety of fabrication problems, associated with core hardware procurement, can be expected to require technical assistance and experimental effort to resolve. These demanded considerable attention in CY 1964 and items such as TaC loaded elements and substitute materials for the existing control plates have already arisen for consideration early in 1965.

D. Irradiation Effects

1. NERVA Structural Materials Irradiation Program

Introduction - NERVA structural materials will be subjected to neutron irradiation at extreme temperatures at high dose rates to integrated fluxes of 1×10^{16} to 5×10^{18} nvt during a 20 minute full power test. In addition, decay heating after shutdown will cause significant temperature excursions in materials around the core. The object of the structural materials irradiation program is to define property changes in materials subjected to this complex environment and thus increase reliability of the engine. Also, data gathered in environments more severe than NERVA may permit increases in performance levels to which conservative limits have been set out of ignorance of the combined effects of stress, irradiation and temperature.

Materials Selection - In selecting materials for irradiation experiments, a complete review of all structural components and materials in the engine was conducted. Materials were chosen according to the following criteria:

Critical function - components for which damage or failure would seriously affect engine performance

Possible damage mechanisms - although little is known of materials in a NERVA environment, modes of damage, but not its extent, may be predicted from data in the literature. Similarly, some materials can be safely assumed not to be deleteriously affected by the NERVA environment.

Margin of safety in design - materials will not be irradiated where safety margins greatly exceed the type and extent of damage expected.

Representative alloys - in some cases an alloy representative of a class of materials will be tested in lieu of each individual material.

To avoid unnecessary testing where the same material is subjected to different environments, tests will be performed only under the severest conditions. Also, a literature survey has been conducted to avoid duplication of results and to indicate potential problem areas.

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Proposed CY 1965 Program - The proposed CY 1965 program consists of a series of tests at the Plumbrook Reactor Facility (PBRF) at ambient and elevated temperatures. The relatively high dose rate at PBRF, 10^{13} nv versus 10^{11} nv at GTR, is desirable because of possible dose rate effects. The maximum NERVA dose rate is 10^{15} nv, considerably higher than GTR or PBRF. However, integrated neutron doses equal to or exceeding those of a 20 minute hot test are feasible at Plumbrook, the maximum being 5×10^{18} nvt ($E > 1$ mev) for a 40 hour cycle.

PBRF Elevated Temperature Test Series

Test No. 1

Because of space requirements only a limited number of specimens may be tested at PBRF at one time. The alloys selected for testing at 340°F (171°C) include:

<u>Material</u>	<u>Component</u>
Inconel 718	Tie rod, nozzle end seal
Inconel X-750	Alternate for 718
Ti-5 Al-2.5 Sn	Core band
	Core support ring

These materials have critical support functions in and around the core, and a high degree of reliability is necessary. The tie rods provide the sole vertical support for the fuel elements and the core band creates a positive bundling pressure around the dome end of the core. Failure of KIWI fuel elements has been attributed to insufficient lateral support. The core band is an integral part of the redesigned NERVA lateral support system for alleviation of core buckling and vibration. Being close to the core, tie rods and the core band also see elevated temperatures and a high neutron flux. For these reasons Inconel X-750, Inconel 718 and Ti-5 Al-2.5 Sn are given prime consideration. This test, in addition to a series of irradiation tests on Inconel X-750 and 718 at cryogenic and ambient temperatures, will yield valuable insights to the behavior of face-centered-cubic, precipitation-hardened super alloys in radiation fields. This alloy class is of particular importance to NERVA because of its general strength at high temperature, corrosion resistance and toughness at cryogenic temperatures.

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Tensile and notch tensile data of the irradiated specimens will be obtained at: 80°F (27°C), appropriate decay heating temperature, and 80°F (27°C) after annealing at the decay-heating temperature. Electrical resistivity measurements and transmission electron microscopy will be performed on Inconel X-750 and Inconel 718 to study damage mechanisms and annealing kinetics.

Test No. 2

A significant problem may exist with irradiation-induced growth in the pyrolytic graphite insulating sleeves. Because of its good across-grain thermal insulation characteristics, pyrolytic graphite is used for insulating tie rods from the very high core temperatures. While the true thermal conductivity of pyrolytic graphite is expected to decrease under neutron irradiation, irradiation growth may cause partial closure of the delaminations in the insulating sleeves and result in an increase in the apparent conductivity. This may cause an increase in tie rod temperature. Ambient and elevated temperature irradiations, required because of the steep thermal gradients across the sleeves in service, would yield the required data. Thermal conductivity, dimensional changes and lattice parameter changes measured on a pre-post basis would permit prediction of the behavior of liner tubes in a NERVA environment.

If the irradiation capsule size and design permit, PBRF Test No. 2 will include NbC-coated ATJ graphite specimens. This material is used for NERVA support blocks. Radiation-induced creep of graphite is a recognized phenomenon. Differential creep of the NbC coating and the ATJ graphite may cause cracking of the coating which would then result in severe hydrogen corrosion of the NERVA support block. A test of this nature would include examination of coating adherence and pre-post hydrogen corrosion testing. Examination of the mechanical behavior of the specimens would be contingent upon the institution of a non-irradiation program on the mechanical properties of NbC-coated ATJ graphite.

Test No. 3 - (Ambient Test)

This test will complete the series of cryogenic, ambient and elevated temperature tests on Inconel X-750 and Inconel 718. The same type of post-irradiation data will be obtained

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as that in PBRF Test No. 1. Depending upon the results of the cryogenic irradiation of Ti-5 Al-2.5 Sn, this test may be expanded to include the titanium alloy. Should space be available in the irradiation capsule, a problem survey will be conducted on the Inconel alloys to investigate possible synergistic effects of stressing during irradiation. Limited data indicated stressing in-pile greatly increases irradiation damage.

Test No. 4

Serious problems exist with radiation effects on organic materials. Therefore, the adhesives used in the NERVA engine should be evaluated, for most of them belong to this class of materials. Suitable radiation-resistant cements must be found for applications such as the following:

- 1) Joining of orifices to fuel elements.
- 2) Cementing of instrumentation.
- 3) Joining graphite for segmented inner reflectors.

A 500°F irradiation of NERVA adhesives at PBRF is proposed for inclusion in the CY 1965 irradiation program. The table lists the types of adhesives and applications, in order of decreasing urgency, on which radiation effects data is required.

<u>Adhesive</u>	<u>Application</u>
RTV 891 (Silicone)	Orifice cement
Imidite (Polyimide)	Orifice cement and segmented barrel cement
Varn 32102NJ (polyamide-imide varnish)	Orifice cement and segmented barrel cement
Poly dibenzothiazole and various modifications	Segmented barrel cement
P514 (three conditions): As cured As carbonized As graphitized	Segmented barrel cement instrumentation cement
M754	Instrumentation cement filler strip cement segmented barrel cement

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<u>Adhesive</u>	<u>Application</u>
National Carbon No. 14	Instrumentation cement
Various ceramic cements such as Bean H	Instrumentation cements

The number of adhesives to be included in this test is dependent upon specimen size and geometry. In addition to this irradiation, other tests at ambient and cryogenic temperatures are required. From initial results, testing of modifications of the above materials and other materials may be warranted at the same temperature of irradiation as the first adhesives test. Also, inorganic instrumentation cements should be investigated as this class of adhesives is expected to sustain less radiation damage than organic cements.

Problem Surveys - In addition to the formal CY 1965 program it is proposed that compact irradiation tests be planned in the event that space becomes available in non-materials tests. These tests could be used to evaluate potential problem areas (problem surveys) and indicate directions for further tests. Three areas where such tests would be particularly applicable are: radiation-induced stress relaxation of springs, synergistic effects of stress and temperature on radiation damage, and stored energy effects in graphite.

Projected Program - This paragraph is included to indicate possible directions for expansion of the CY 1965 program. The table lists the components, materials, temperatures and properties for which radiation effects information is desired.

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<u>Material</u>	<u>Component</u>	<u>Radiation Temp.</u>	<u>Properties</u>
H4LM Graphite and other candidate inner reflector materials	Inner Reflector	Ambient	Thermal Conductivity Elastic Modulus Strength
Ti-5 Al-2.5 Sn	Core Support Ring	Ambient	Ductility H ₂ embrittlement
ZTA Graphite	Seal Segments	Ambient	Thermal Conductivity Strength Elastic Modulus
Invar and Ni-Span-C	Core Band	Elevated	Ductility, strength, thermal expansion characteristics
TDM Nickel	Tie Rod	Ambient	Strength
Instrumentation Brazes (primarily silver solders)	Instrumentation Leads	Ambient	Structural, electrical and sealing joint efficiencies

Because of the very high NERVA dose rate, a problem survey of the effect of dose rate on radiation damage is also desired. Cryogenic and elevated temperature irradiation of tie rod materials at a high dose rate would yield this much needed data.

Additional tests will be dictated by the results of the proposed tests. Also, as the ultimate objective of the irradiation program is the determination of radiation effects up to a 60 minute NERVA dose, further testing must be done at higher doses under the same conditions as earlier tests.

2. Post-Operation Materials Evaluation

Progress in an engineering development program such as NERVA is dependent on continuing tests of components and assemblies as they evolve from prototypes through various modifications to increasingly efficient, reliable, and versatile products. Obtaining the required contribution from each test depends in turn upon careful planning to derive the maximum information possible from it. One source of test data is the examination, destructively and non-destructively, of the condition of various materials after test. In the case of irradiation effects, the test conditions cannot be simulated exactly by exposure of components in materials test reactors. In obtaining such data, the Materials Department works in concert with other

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departments of WANL, as well as with other personnel at NTS and LASL. The procedures to be followed in post-operation examination of NRX-A1, A2 and A3 have been presented in the respective Test Plans and Test Specifications, to which references should be made for detailed information as to work plans in this area during CY 1965. Similar documents will be prepared for subsequent scheduled tests. It should be pointed out that a large portion of the post-operation determination of material properties has been assigned to the Materials Department Laboratories, at least in the case of NRX-A2, and tentatively in the case of NRX-A3 also. Some examinations which it has been possible to forego for NRX-A2 and A3 can not longer be dispensed with for the NRX-A4 test, and a larger post-operative program will then be needed.

E. Specifications

1. New Materials, Process, and Equipment Specifications

Preparation of new material, process, and equipment specifications will continue to require a high level of effort. The supplier acceptance portion of the program, initiated in CY 1964 will probably be discontinued because of reduction in manpower assigned to this function.

2. Revisions to Existing Specifications

A continuing high level of effort will be required to improve existing specifications. Supplier and customer comments, experience with core hardware, test results, materials development data, and changing design requirements will form the basis for these revisions.

3. Equipment Specifications

a) Accumulation of data on existing equipment specifications will be completed and a numerical compilation issued.

b) Existing equipment specifications will be written in a standard format as they come up for technical revision.

4. Compilation of WANL Specifications

Bi-weekly listings of new specifications and semi-annual compilations of WANL specifications will be issued. A cross-index according to material, form, and process is needed

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as a reference for designers and others who use WANL specifications. This will be combined with the semi-annual compilation.

6. Government Specifications and Standards

Surveillance of government specifications and standards will be improved by adopting the subscription plan recently offered by the Department of Defense.

7. Design Review and Drawing Sign-Off

Design reviews to uncover potential problems and drawing approvals of materials and processes will continue to require a significant effort from the materials and processes standpoint.

8. Data Retrieval

Investigation will be made of the feasibility of putting materials and specification data on tape for use in a computer to facilitate information retrieval. Data would be obtained from such sources as supplier approval requests and publications. It would be useful to Reactor Mechanical Design, Reactor Analysis, and the Materials Department in their analysis of design and material problems.

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APPENDIX A

PROPOSED CY 1965 WORK PROGRAM NON-FUEL STRUCTURAL MATERIALS

A. Vendor Liaison, Vendor Evaluation, and Consulting	24 Man months
B. Materials Development, Evaluation, Application and Behavior	66 Man months
1. Materials Data Sheets	
2. Graphite Studies	
a) Improved inner reflector materials	
b) Evaluation of promising new structural graphites	
c) Annealing of pyrolytic graphite	
d) Pyrolytic graphite alternate materials	
e) Hydrogen corrosion of uncoated graphite	
f) Hydrogen corrosion of NbC coated support blocks	
g) Permeability and impregnation of new inner reflector cylinder materials	
3. Beryllium Components	
a) Mechanical and physical properties	
b) Thermal shock behavior	
c) Aging of beryllium	
4. Hydrogen Environmental Effects on Titanium Alloys	
5. Nickel Base Alloy Components	
a) Inconel type alloys	
b) Dispersion-hardened alloys	
6. Seizing and Galling of Components	
7. Thermal Capsules	
8. Instrumentation Materials Problems	
9. Other Materials Problems (Miscellaneous Materials)	
C. Fabrication Development	30 Man months
1. Joining of Graphite to Itself and to Other Materials	
a) Carbonaceous cements	
b) High-temperature organic structural cements	
c) Ceramic adhesives	
2. Control Drum Plate Assembly Fabrication - Welding	

Appendix A (Continued)

3. Process Development	
4. Miscellaneous Fabrication Problems	
D. Irradiation Effects	18 Man months
1. NERVA Structural Materials Irradiation Program	
2. Post-operation Materials Evaluation	
E. Specifications	42 Man months
1. Equipment Specifications	
2. New Materials, Process and Equipment Specifications	
3. Revisions to Existing Specifications	
4. Compilation of WANL Specifications	
5. Government Specifications and Standards	
6. Design Review and Drawing Sign-Off	
TOTAL	180 Man months

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APPENDIX B

TME'S ISSUED DURING CY 1964 TO AUGUST 15, 1964 FOR PROJECT 8910

<u>TME No.</u>		<u>Date of Report</u>
319	Selection of NRX-A Tie Rod Materials	10/17/63
515	Metallurgical Aspects of the Reuse of Beryllium Reflectors	9/12/63
563	Metallurgical Evaluation of the NERVA NRX-A Tie Rod Fabrication Process	11/ 1/63
567	Evaluation of Three Candidate Cluster Plate Materials	10/18/63
571	Evaluation of Orifice Cements for NERVA	2/14/64
590	Fabrication Development of Shielding Material	11/ 8/63
611	Material Selection for the NRX-A Core Support Barrel	12/ 4/63
624	Development of a Brazing Process for Seal Blocks	12/17/63
625	Evaluation of an Electron Beam Welding Process for Control Drums	12/17/63
626	Evaluation of Resistance (Capacitor Discharge) Welding for Attaching Instrumentation	12/17/63
628	Development of a Gold Plating Process to Withstand a 1350°F Brazing Cycle	12/17/63
649	Evaluation of Plasma-Sprayed Pyrolytic Graphite Components	1/10/64
652	Irradiation Behavior of NERVA Shield Capsules	1/10/64
654	Aluminum-Boron Control Plates for the NERVA Reactors: Fabrication and Properties	1/13/64
672	Gadolinium-Stainless Steel Development - Part I	2/ 3/64
679	Gadolinium-Stainless Steel Development - Part II	2/ 7/64
705	Irradiation Effects on Tie Rod Materials	2/28/64
714	Evaluation of Ni-SPAN-C 902 for NERVA Fasteners	3/18/64
738	Investigation of Titanium Bolt Failures	4/ 2/64
741	Survey of Graphite Materials for NRX-B Module Tubes	4/17/64
755	Evaluation of Thoria Dispersion-Hardened Nickel for NERVA Applications	4/27/64

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Appendix B (Continued)

765	Evaluation of NRX-A2 Type Inner Reflector Properties	5/ 8/64
827	Thermal Capsule Calibrations	6/30/64
831	Properties of Fiber Based Graphites	8/10/64
847	Joining of Graphite Fuel Elements for NRX-B Advanced Design	7/15/64
876	Potting of Thermal Capsules and Thermocouples in the NRX-A Reactor	7/15/64
891	Structural Graphites with High Thermal Expansion	8/15/64

REPORTS TO BE ISSUED DURING BALANCE OF CY 1964

- 1) Evaluation of Pyrolytic Graphite Used in NERVA Reactors
- 2) Properties of ATJ Graphite
- 3) Evaluation of Coated Support Blocks
- 4) Justification for the Use of Inconel 719 for Tie Rods
- 5) Heat Treatment and Dimensional Stability of Large Cylinders of 2219 Aluminum Alloy
- 6) Evaluation of Metallurgical Processes for Fabrication of Aluminum Core Support Barrel
- 7) Effect of Heat Treating and Brazing Processes on Instrumentation
- 8) TIG Spot Welding Stainless Steel

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