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Westinghouse Astronuclear Laboratory

REACTOR NON-FUEL MATERIALS PROGRAM FOR C. Y. 1966
(Title Unclassified)

E. L. Layland
Materials Department
July 14, 1965

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REACTOR NON-FUEL MATERIALS PROGRAM FOR C. Y. 1966

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INTRODUCTION

The proposed Reactor Non-Fuel Materials Program for C. Y. 1966 provides only the most essential materials support to the NERVA effort. The day-to-day contacts with personnel in other departments such as Reactor Mechanical Design, Manufacturing, Quality Assurance, Reactor Analysis, Control Development, and Purchasing, together with liaison with vendors, and with SNPO and AGC provide the basis for formulating the proposed C. Y. 1966 Work Program. In C. Y. 1965, the level of effort was 15.3 engineers, 9 technicians, and \$120,000 for materials and services. During the year, the manpower was bolstered by 2 engineers supported by M&S funds to improve the corrosion resistance of support blocks. The C. Y. 1965 effort was entirely a hardware support program geared to the materials aspects of problems in procurement, processing, assembly, and reactor performance. The proposed C. Y. 1966 program entails 15.3 engineers, 9 technicians, and \$72,000 for materials and services. See Table I for the allocation of effort. This funding will provide the same type of support as in C. Y. 1965 but at the indicated lower level of effort. It will provide materials evaluation but not materials development. It will not provide a desirable level of effort in such areas as: determination of the physical properties of NERVA materials as affected by temperature; determination of irradiation effects on metals, graphites and organic materials; development of a high-temperature Station 45 thermocouple; development of a high-temperature orifice sealant; and parametric studies of hydrogen corrosion of graphite.

In past years, the proposed Work Program has included a lengthy summary of the previous years' accomplishments. This year, the summary has not been included because the Quarterly Progress Report and Annual Report as well as the reporting of data via TME's and other correspondence have adequately documented the program.

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NRX-A HARDWARE SUPPORT

A. Specifications

The preparation of new material, process, and equipment specifications and revision of existing specifications will require the same level of effort that existed at the end of C. Y. 1965. Supplier and customer comments, experience with core hardware, test results, materials development data, and changing design requirements will form the basis for these revisions. Bi-weekly listings of new specifications and semi-annual compilations of WANL specifications will be issued. A cross-index according to material, form, process, and NERVA application is needed as a reference for designers and others who use WANL specifications. This will be combined with the semi-annual compilation. Surveillance of government specifications and standards will be improved by adopting the subscription plan recently offered by the Department of Defense.

B. Drawing Review and Approval

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.

C. Variation Requests, Approval Requests, and Engineering Change Orders

Considerable effort will be required, as in the past, to evaluate from a materials and processes viewpoint variation requests and approval requests from vendors and engineering change orders from the design groups.

D. Vendor Liaison

Vendor liaison and vendor evaluation will continue in an effort to improve the NERVA product and the delivery and, wherever possible, the cost. Although vendors of essentially all materials used in NERVA have been contacted, continuing effort is necessary with emphasis still 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 to continue evaluating manufacturers for progress in solving present problems, to improve specifications as vendors'

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progress permits, to help develop better products and to examine new products which may do existing jobs better.

E. Consulting

Consulting will continue to be a major effort, for numerous requests for advice on materials and processing come from design, manufacturing, quality assurance, purchasing and suppliers' personnel. Typically, help is needed on selection of materials, the properties of materials in the NERVA environment, fabricability of materials, methods of evaluation of materials, and determination of those properties needed in specifications. Also, the urgent 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.

F. Trouble Shooting

During hardware procurement and assembly, unexpected materials failures and damage occasionally occur. These require immediate action to determine the cause of the difficulty and remedial measures. Recent examples include the A-3 reactor temperature excursion with resultant overheating of core components, the A-3 central element contamination problem, cracks in the heads of tie rods, premature failure of a nozzle end seal ring during pressure testing and breakage of titanium bolts during a vibration test.

G. Materials Evaluation

The continued advances in reactor performance 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 needed. 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, and study of failure mechanisms of materials of potential interest.

1. Reactor Post-Operative Examination

Progress in an engineering development program such as NERVA is dependent on continuing tests of materials, 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 so post-operative examination of the materials in the NERVA reactor are essential. In obtaining such data, the Materials Department works in concert with other departments of WANL, as well as with other personnel at NTS and LASL. The procedures to be followed in post-operation examination of NRX-A4 and A5 are presented in the Test Plans and Test Specifications, to which reference should be made for detailed information as to work plans in this area during C. Y. 1966. 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.

2. Hydrogen Corrosion of 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, corrosion rates will be established as a function of temperature to 3000°C and pressure to 1400 psi 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 protective 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 deposition of corrosion products from the hydrogen gas will be examined. Dimensional and weight change will be the primary methods of evaluation of 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.

3. Instrumentation Materials

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 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 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 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. Planned experimental work includes:

- a. Continuation of chemical compatibility studies between electrical insulators, such as BeO , ThO_2 and MgO , and refractory metal sheaths or wires in order to select combinations of materials for high-temperature thermocouples that will survive the temperature levels without catastrophic interaction,
- b. Metallurgical examination of experimental and qualification-type high-temperature thermocouples and,
- c. Continuation of investigation of protective coatings for high-temperature thermocouple sheaths to avoid catastrophic melting due to metal-carbon eutectic reactions.

4. NERVA Materials Properties Data Book

Work will continue in the preparation of data sheets for inclusion in the NERVA Materials Properties Data Book. Specific properties for many materials are still needed to assure reliable designs and justifications for particular selections. For example, statistically significant physical 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. For structural graphites and many metals, conductivity, tensile strength, flexural strength, fatigue strength, Young's modulus, coefficient of thermal expansion, Poisson's ratio, creep and thermal shock resistance are needed.

5. Materials Irradiation

Structural materials will be subjected to neutron irradiation at extreme temperatures at high dose rates to integrated fluxes of 3×10^{16} to 1.5×10^{19} nvt during a NERVA one-hour, 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 reactor. 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.

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

Critical function - components which would seriously affect engine performance if damaged.

Possible damage mechanisms - although little is known of materials in a NERVA environment, modes of damage, but not extent of damage, 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 most severe conditions. Also, a literature survey has been conducted to avoid duplication of results and to indicate potential problem areas. The emphasis will be placed on problems demonstrated by previous materials irradiation tests and the results of NRX-A2 and A3 reactor runs. Other important, but less urgent, problem areas must remain untouched. With the completion of irradiation tests 37/W001 and 37/W401 at the GTR (simulating up to a 20-minute NERVA full-power test), the materials of particular concern have been identified as hot-pressed block beryllium, the titanium - 5% aluminum-2 1/2% tin alloy, and Inconel 718 alloy weldments. Because of the susceptibility of organic materials to irradiation damage, much data is available in this field. With the rapid development of new organic materials, however, future tests are required to evaluate them. During the NRX-A2 EP IV test, the graphite inner reflector never reached steady-state temperature. The continually rising temperature is believed to be due to a decrease in the thermal conductivity of the graphite as a result of radiation damage.

Since emphasis will again be placed on cryogenic irradiation effects, both the GTR and PBRF reactors will be utilized. The PBRF gaseous helium loop is

necessary for metallic materials because of the required high neutron doses. Graphites and organics will be irradiated at the GTR reactor where the large testing space is a distinct advantage. Results of WANL test No. 37/W401 demonstrate that cryogenic testing after cryogenic irradiation must be performed without warm-up because, in almost every instance, annealing of damage proceeds rapidly at room temperature and below. Since no cold transfer or in-pile testing technique has been funded for the PBRF helium loop, the number and types of tests cannot be fully presented. Several cold transfer and in-pile methods are now being studied. It is essential that some method be funded to permit further study of this serious problem.

Test No. 1: Graphite Thermal Conductivity and Organic Materials Tests

In-pile thermal conductivity measurements on H4LM inner reflector graphite and P-03 high-strength, isotropic graphite will be accomplished at the GTR with equipment supplied by AGC. Thermal conductivities will be measured on a pre-post basis at -320°F after irradiation at -320°F . While the measurements will be made in pile, they cannot be made while the reactor is at power because of prohibitive gamma heating and background noise. The effects of annealing will also be studied with the range of temperatures being determined by the equipment capability. Since the thermal conductivity apparatus has not been proven in an irradiation field, the planned 200-hour test will consist of a 50-hour and a 150-hour test. After 50 hours at power, the reactor will be shut down, measurements of thermal conductivity will be made, and the apparatus will be disassembled and inspected. Barring damage, it will be reassembled and irradiated for 150 additional hours. Total integrated neutron dose will be on the order of 5×10^{17} nvt ($E > 1 \text{ Mev}$).

Advantage will be taken of the large irradiation space at GTR by including organic materials in the test. Candidate materials are: a new higher-temperature thermo-plastic polymer, known as PPO (polyphenylene oxide) which may be suitable

as a NERVA adhesive; a new, high-strength, carbon-filled silicone rubber, known as DC 92-018, which may be suitable as a NERVA solvent/adhesive; a new metal oxide-filled silicone grease possessing high thermal conductivity which may be suited as a potting compound in instrumentation transducers to dissipate gamma heating; and a 1000°F (538°C) aromatic polymer for adhesive applications.

Test No. 2: Elevated Temperature Irradiation of Inconel 718

It is planned to irradiate Inconel 718 tie rod material tensile specimens at 440°F to an integrated dose of approximately 2×10^{19} nvt ($E > 1$ Mev). Depending on further analysis of the 400-hour cryogenic irradiation at GTR (37/W401), Inconel X750 specimens may be included in the test. At this tie rod maximum service temperature, the material may suffer irradiation-induced weakening and /or embrittlement. Possible mechanisms of damage at this temperature include irradiation-induced additional precipitation of the strengthening phases, irradiation-induced solution of the previously formed precipitate, and the temperature-enhanced diffusion and agglomeration of helium resulting from the boron-neutron reaction.

Test No. 3: Cryogenic Irradiation Effects on NERVA Alloys

A number of materials in test No. 37/W401 at GTR exhibited severe cryogenic irradiation damage at simulated NERVA doses approximating 5-20 minutes of full-power operation. To insure the reliability of future reactor tests, it is essential to perform additional cryogenic irradiations at higher integrated doses, particularly on the alloys shown to be severely affected. The cryogenic helium gas loop being constructed at PBRF will be the only facility available where the required neutron doses ($\approx 10^{19}$ nvt $E > 1$ Mev) can be practically obtained at cryogenic temperatures. It is proposed to utilize this loop to determine cryogenic irradiation damage to the tensile properties of the following materials. Probably only one material can be tested during each irradiation, thus necessitating several reactor cycles to irradiate all of the high-priority materials.

A. High Priority

1. Beryllium
2. Ti-5Al-2.5 Sn Eli
3. Inconel 718
4. Inconel 718 weldments

B. Lower Priority

1. Al-2219-T852
2. Al-2219-T81
3. AISI 303 Se annealed
4. AISI 301 full hard
5. Inconel X-750

6. Support Block Improvement

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 dependent upon the quality control type evaluation of the coating on support blocks to determine performance in the design environment. Corrosion tests with coated sections of support blocks will be run and destructive metallographic examinations of coating thickness will be made. These tests will be conducted in conjunction with fuel element corrosion tests. This effort will involve interpretation of hydrogen corrosion test results on process samples. Information derived from this study will be fed back and factored into coating processes and quality control.

7. Graphite Joining

The application of cementing as a graphite joining technique has received considerable attention. This work will be continued to solve problems arising from design modifications. The carbonaceous cements have been used as a means of

joining filler block keys and thermal capsule containers to fuel elements. Other cement formulations will be evaluated to decrease the inherent porosity of carbonaceous cements after carbonization and graphitization. Recent work in polymer chemistry has yielded 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; this type of cement may be more resistant to irradiation, since aromatic polymers are generally more stable than aliphatic polymers. They can be used in a variety of applications if they have sufficient resistance to irradiation. Some applications, which will be investigated, are joining of instrumentation to the graphite inner reflector; repair of the inner reflector; and sealing and joining of graphite segments for fabrication of a multi-segmented inner reflector barrel for future core design.

Brazing of graphite has been developed for non-NERVA use and has the advantages of high-strength, impermeable joints. During the latter part of C. Y. 1965, graphite brazing is being studied and will be continued in C. Y. 1966 for specific application to a new keyed-fuel element, support blockless cluster design and to the sealing of stainless steel orifice jets in fuel elements.

Because of their high-temperature capabilities, inorganic adhesives are being considered for NERVA application in spite of their shortcomings, i. e., the glassy-type adhesives must be melted (or matured) at elevated temperatures and must contain carbide-forming compounds in order to wet graphite; they are inherently brittle; they are susceptible to thermal shock unless the adhesive layer is extremely thin; and those containing water in the "as-cured" state fail by the loss of water of hydration. Some work at the Westinghouse Research Laboratories has shown promise for sealing and joining and will be investigated further.

8. Control Materials

Control plate assemblies for the NERVA reactors are being produced by the WANL Materials Department. Adaptation of the LASL fabrication process was accomplished 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.

9. Evaluation of Promising New Structural Graphites

The demand for increased reliability in 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 with reactor design requirements. The strongest structural graphite now available as a result of evaluation is an isostatically molded grade, known as P-03, having exceptionally high strength as follows: tensile strength - 5,500 psi; flexural strength - 8,000 psi; and compressive strength - 20,000 psi. More recently, graphites have been produced with high coefficients of thermal expansion, matching that of the niobium carbide protective coating for graphite. Also, new fiber-base and filament-wound graphites are becoming available and will be investigated.

10. Welding Close Tolerance Assemblies

The increased use of electron beam welding for critical reactor components such as tie rods has generated a need to study weld tooling concepts for electron beam processes on the basis of specific applications as they arise. The effort will entail the potential modification of the tie rods, flow screens and other similar close tolerance structures.

11. Fabrication of New Transducer Structures

The use of short immersion thermocouples for support blocks and exit gas temperature measurements involves complex structures in the area of the tie bolt termination.

Development of fabrication and assembly processes will be required.

TABLE I

FUNDING OF C. Y. 1966 NON-FUEL MATERIALS PROGRAM

	<u>Engineer Man-Months</u>	<u>Technician Man-Months</u>	<u>Materials & Services - \$</u>
A. Specifications	35	6	----
B. Drawing Review and Approval	5	--	----
C. Variation Requests, Approval Requests and Engineering Change Orders	2	--	----
D. Vendor Liaison	4	--	----
E. Consulting	48	--	----
F. Trouble Shooting	4	6	3,000
G. Materials Evaluation			
1. Reactor Post Operative Exam.	10	8	5,000
2. Hydrogen Corrosion of Graphite	14	16	12,000
3. Instrumentation Materials	10	18	15,000
4. NERVA Materials Data Book	4	6	----
5. Materials Irradiation	14	15	20,000
6. Support Block Improvement	12	6	4,000
7. Graphite Joining	9	15	3,000
8. Control Materials	--	--	----
9. Evaluation of New Graphites	6	6	8,000
10. Welding Close Tolerance Assemblies	3	3	1,000
11. Fabrication of New Transducers	<u>3</u>	<u>3</u>	<u>1,000</u>
Total	183	108	\$72,000