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REACTOR NON-FUEL
MATERIALS PROGRAM
C.Y. 1963 & C.Y. 1964

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WORK PROGRAM FOR REACTOR
NON-FUEL MATERIALS DEVELOPMENT AND EVALUATION

NE 1312 - CY '63
NE 8910 - CY '64

I. Summary NE 1312: October, 1962 - June, 1963

The CY '63 efforts to date have been concentrated on the preparation and issuance of urgently needed material and process specifications; consultation with design, manufacturing, quality control and purchasing personnel in the selection of materials; acquisition of materials properties data for the NERVA Materials Manual; liaison with suppliers; and the initiation of planning for the non-fuel materials development program.

A. Vendor Liaison, Consultation, Evaluation and Specifications

1) Specifications:

In response to the urgent need for materials and process specifications, approximately 50 WANL materials specifications and 45 process specifications were written and issued. A weekly listing of new specifications and quarterly compilations of all WANL specifications are now issued.

Tooling up for the specification activity has involved acquisition of specifications from other agencies, such as:

- (1) Westinghouse Corporate Purchasing Department Specification
(Sheets and cards)
- (2) Westinghouse Corporate Process Specifications
- (3) Westinghouse Corporate Materials Handbook
- (4) Westinghouse Corporate Design Handbook
- (5) Westinghouse Corporate Safe Practice Data Sheets
- (6) ASTM Specifications
- (7) Federal Specifications

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- (8) MIL Specifications
- (9) A/N Specifications
- (10) SAE (AMS) Specifications

Review of engineering drawings and the selection and approval of specifications for the fabrication of reactor components and ground support equipment continues to be a major effort.

2) Material Data Sheets:

Data acquisition has been heavily emphasized to provide materials properties information for design purposes. Sources have included the literature and suppliers' information, with only a small amount being established experimentally because of expediency and the availability of manpower and test facilities. These data have been issued as 284 data sheets in the form of curves relating the various properties to test temperatures. The data are identified as typical (average), minimum, estimated, and authoritative.

An analysis of NERVA components was made to define the significant mechanical and physical properties involved for reliable design. An accounting of each material considered for each component was made indicating the data available and the data yet to be acquired. Test priorities were established so that the most urgently needed data would be obtained first.

3) Consulting:

Consulting is a large and continuing effort to support the requests from design, manufacturing, quality control, purchasing, and suppliers' personnel. Typically, help is needed on selection of materials, the properties of materials in specific environments, fabricability of materials, methods of evaluation of materials determination of those properties needed for specifications, evaluation of suppliers, etc. Actually, consulting is involved in each of the work areas described below.

B. Material Evaluation, Application and Behavior

1) Graphite Components:

Each of the five pyrolytic graphite manufacturers (High Temperature

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Materials, Incorporated; Raytheon Company; Beryllium Corporation; General Electric Company; and Super-Temp Corporation) was visited to evaluate their capabilities of meeting WANL requirements. These visits served also to explain our requirements to the suppliers to facilitate procurement of satisfactory material. The Southern Research Institute was chosen as sole source agent for final testing acceptance (principally thermal conductivity testing) of pyrolytic graphite components. Also development of an in-house method of thermal conductivity testing to 3200 °F was begun. Recent results indicate that the specified requirements for across-grain thermal conductivity of tubes and plates are obtainable.

A comparison of available material properties with the desired design properties for several recently designed reactor components resulted in the recommendation of several grades of graphite most suited for the specific applications. Furthermore, a re-evaluation of the design requirement for Inner Reflector cylinders resulted in a search for better grades of graphite than are presently being procured.

The carbide coatings on regular support blocks (coated by High Temperature Materials and by the Westinghouse Astrofuel Facility) are being evaluated.

2) Tie Rod Materials:

To find a suitable material for the tie rods, eleven promising materials were reviewed from the standpoint of mechanical properties, fabricability and irradiation susceptibility. From these, Inconel X-750 emerged as the primary material believed suitable for the application and Inconel 718 was chosen as a back-up material for even greater potential because of its higher yield and tensile strengths. Fabrication development with the Hunter Company has resulted in experimental tie rods for metallurgical and dimensional evaluation now being conducted. The procedure involves hot upsetting both ends of the rod followed by hot forging the head to finished shape, and cold rolling of the threads. Nearly completed studies have shown that both materials and fabricating procedures are satisfactory for regular rods. A report has been prepared and will be issued.

3) Titanium Support Rings and Tie Bolts:

The titanium alloy containing 5% aluminum, 2-1/2% tin, and extra low interstitials was chosen for the forged core support ring, bottom support ring, and outer reflector dome support ring because of its retention of adequate ductility at cryogenic temperatures. Because of the unusual care required for titanium forging, considerable effort was necessary to evaluate vendors' facilities and procedures and to "trouble shoot" on production forgings.

4) Lithium Hydride Shield Pellets:

Methods were developed to compact lithium hydride powder by cold pressing and heat treating (sintering). Suitable heat treatment minimizes outgassing and samples have been encapsulated in stainless steel containers for irradiation testing. Samples have been irradiated and are now being evaluated.

5) Beryllium Components:

Most effort to date has been data accumulation and vendor liaison. However, recently an experimental program was begun to study the effect of various machine finishes and surface chemical treatments on the tensile properties. The powder metallurgy beryllium products (VHP) being used are brittle and as a result their observed strength is affected by surface defects and conditions.

6) Cluster Plate Materials:

Because of gadolinium's high neutron capture cross section, it has been alloyed with type 304 stainless steel in an experimental material for evaluation as cluster plates, which are needed to prevent flux peaking at the top of the fuel elements. Inhomogeneity and poor fabricability of the material emphasize the need for processing development. In the interim, a silver-base alloy containing indium and cadmium has been evaluated and selected as an alternate material.

7) Seizing and Galling Prevention:

A form of nitriding heat treatment, known as Tufftriding, has been evaluated as satisfactory for obtaining a thin, hard, galling-resistant surface on stainless steel components. A Process Specification has been prepared. Anti-galling treatments for

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titanium being investigated include surface chemical conversion coatings and nickel, gold, and silver electroplating, as well as as dry film lubricants.

C. Fabrication Development

1) Control Drum Plate Assembly Fabrication Development:

The control drum plates are made of a composite powder metallurgy product comprising a dispersion of $-100 + 325$ - mesh boron powder in an aluminum matrix. Because of the large amount of dispersed nonmetallic phase, and the need for homogeneity, an experimental development effort was required to evolve specifications for processing and purchasing of these plates and the plate assembly. Previous work of a similar nature carried out at LASL was reviewed and utilized where applicable. Suitable equipment was made and procured, and satisfactory procedures were established for the manufacture of these plates, from the raw materials through to the completed assembly. The production of control drum plates for deliverable hardware has been accomplished.

2) Process Development - Joining (Brazed Instrumentation Pass-through):

The sealing of the instrumentation leads is accomplished by brazing the leads in groups of from seventy to one-hundred, into a one inch diameter plug drilled with a corresponding number of holes. The close spacing of the brazed joints presents many heating and alloy flow problems. Fluxes cannot be used since they cannot be reliably removed. The iron-constantan and tungsten-tungsten rhenium thermocouples are damaged by excessive brazing temperatures so the brazing temperatures must be restricted to 1350°F . Therefore, reducing gasses such as hydrogen can not be used. The braze must be produced within one inch of a cylindrical sealing surface that has been previously ground to $\pm .0005$ inch tolerance. Finally, the brazing process must be carried out as part of the reactor assembly operations. Microscopically sound test joints have been produced using a self-fluxing braze material that is essentially a low melting silver braze with a small amount of phosphorous. This material will, without flux, wet either copper, gold or silver. Accordingly, a process and design have been developed for converting the surface of the pass-through plug to copper by inlaying a copper disc

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in a recess in the plug prior to drilling the necessary holes in the pass-through. A process has been developed for plating the brazing area of the stainless steel instrument leads with gold. A portable induction unit with an inert atmosphere and a graphite susceptor has been employed in making the test joint. A change in plug diameter has necessitated a redesign of the induction coil. The redesigned unit will be used to produce pressure test samples and for production brazing.

3) Process Development - Joining (Control Drum Assembly Welds)

As a final operation in the assembly of control drums the caps must be welded to the outer cylinder. The weld must be sound and must form a seal between the cap and cylinder. The joint should carry up to 50 lb/inch loads. The welding must be accomplished on heat treated (T6) 6061 aluminum alloy within 0.100 inch of a finish machined seal band extending from the rim of the cap. The band must remain intact and no machining is permitted after welding. Autogenous welds have been produced by high voltage (120 KV) electron beam techniques. The welds met all requirements except that one concerned with soundness. In addition, the work surface precluded adequate post weld inspection. The types of defects suggested a lack of weld metal ductility. Accordingly, tests are being run using filler wire and a "V" groove. This combination is expected to increase ductility, produce a smoother weld for inspection, and, additionally, guarantee a certain, predictable weld cross-section to assure the load-carrying ability of the joint.

D. Radiation Effects Program

An irradiation effects program has been prepared to acquire the data needed for the non-fuel materials comprising the NERVA reactor. A program will be proposed for AGC and SNPO approval. This program is based on the irradiation of specimens of the materials with conditions of heat treatment and fabrication similar to those to be used in manufacture. Also irradiation is proposed at temperatures no higher than calculated operating temperatures and with dosages equal to calculated values. Dose rate is also considered extremely important and thus the Plumbrook test reactor is highly desirable because of its high flux. For other materials the GTR facility is proposed. The

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program entailed 1300 specimens (including unirradiated controls) for determination of the notched and unnotched tensile properties. The proposed program included irradiation of:

- 1) Tie rod materials (Inconel X-750, Inconel 718, Hastelloy R-235 and W-545)
- 2) Support cone materials (molybdenum TZM and Inconel 718)
- 3) Core support plate material (aluminum 2219 T 852)
- 4) Core support, dome support, nozzle and support ring and tie bolt material (titanium 5 Al, 2-1/2 SN ELI)
- 5) Fuel cluster preload spring, inner reflector preload spring, control drum power spring, and lateral support spring materials (cold reduced stainless 302 and Inconel 718)
- 6) Bottom support block material (graphite ATJ and RVC)
- 7) Lateral support material (graphite ZTA and ATJ)
- 8) Inner reflector material (graphite H4LM)

Limited irradiation test facilities subsequently caused consideration of a reduced amount of testing. Principally, the reduction in extent of testing will affect the number of tests of each material, but not the number of materials to be tested.

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II. NE 1312 Program: July - September, 1963

A. Vendor Liaison, Consultation, Evaluation and Specifications

1) Specifications:

Preparation of new material and process specifications will continue to require a high level of effort. All of the graphite specifications, for both pyrolytic and conventional types, will be improved to facilitate procurement of material possessing the necessary characteristics. More emphasis than in the past will be required to improve existing specifications and to negotiate with prospective suppliers to obtain their acceptance of the specifications. Normally, supplier negotiation would precede issuance of the specifications, but the demands of production schedules have required specifications prior to supplier acceptance. Present trends indicate that design reviews and drawing sign-offs will require a significant effort from the materials and processes standpoint.

2) Supplier Evaluation and Liaison:

As the NERVA project moves increasingly into the manufacture of components, supplier evaluation will become more important. Evaluation entails vendor facilities, personnel and procedures. Visits will be made to vendors of graphite, beryllium, titanium, aluminum, tie rods, cluster plates, etc. Liaison will be maintained with these vendors to provide technical help, and to monitor critical operations.

Close liaison will be maintained with vendors concerning WANL requirements for inner reflector cylinder material. Trips will be made to vendors' plants. Arrangements will be made to get better data on existing grades of graphite and to work towards the development of improved materials for NERVA requirements.

Closer liaison will be established and maintained with all of the conventional graphite vendors in order to buy material that meets NERVA requirements. Close liaison will be maintained with pyrolytic graphite vendors to continue the trend towards the manufacture of high quality insulating components.

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3) Consultation:

Consultation on materials problems with design, manufacturing, quality control, purchasing and vendors will continue to be a major function. Typically, request for consulting help are urgent, are of short notice, require little or no follow-up, are largely unforeseen, and are not amenable to advance planning.

B. Materials Evaluation, Application, and Behavior

1) Data Accumulation:

Although materials data sheets have been issued, they are far from complete. Accordingly, gaps in the data will be filled by searching the literature, contacting suppliers, and experimentally determining this data. To do this in an efficient manner, all design components were reviewed to determine the important material properties requirements, the data available and the data to be obtained. Priorities were assigned so that, where possible, the most important data could be obtained first. Thus, the physical and mechanical properties of the several grades of graphite used for NERVA were assigned top priority.

2) Materials Evaluation:

In addition to the specific materials evaluations listed in the following sections, effort will be made to keep abreast of new materials and processes. When promising developments emerge, information will be obtained from the literature, from the source, and from in-house investigations, when appropriate.

3) Graphite Components:

a) Evaluation of Niobium Carbide Coating Processes for Structural Components:

i) The vendors' niobium carbide coating processes will be evaluated for approval. A protective coating of niobium carbide is required on several types of components (including pyrolytic graphite) to protect them from hydrogen corrosion and erosion in the expected reactor environment of temperature and flux. Before any facility can start production coating of those components, the process must be approved per WANL Specification 30065.

- ii) This approval will result from the evaluation of coated components per WANL Specification 30065 established for this purpose. Several vendors are currently in the process of preparing proposals for qualification and manufacturing.
- b) Evaluation of Pyrolytic Graphite Components:
 - i) At present, pyrolytic graphite components have not been produced to the NERVA long range objectives. The evaluation of these pyrolytic graphite components will foster the development of better processes and/or components, and will result in improvement of the present specifications. This evaluation and comparison between vendors will give a more realistic base for future procurement actions.
 - ii) This evaluation will consist of studying the visual appearance and soundness, the microstructure, and the thermal conductivity as a function of furnace position for all of the types of components made by each manufacturer. Comparisons of quality will also be made between vendors. Evaluation will continue as materials are received from vendors and the information developed will serve as the basis for more detailed work planning for C.Y. 1964.
- c) Tensile Specimen Design for Graphite:
 - i) The aim of this program is to design a tensile specimen which will reflect the true tensile properties of graphite with the minimum amount of data scatter. The inherent mechanical properties of graphite complicate determination of its tensile property. Graphite manufacturers have quoted tensile properties based on specimen designs which gave data having a standard deviation as large as 30-40%. Care will be exercised to ascertain that specimen design does not optimize properties and present a false concept of the true engineering properties.
 - ii) Five specimen designs are being evaluated for the following criteria:
 - 1. Minimum amount of standard deviation in data.
 - 2. Ability to fracture in gauge length.

iii) Evaluation of the specimens will be completed by August 15, 1963.

d) Graphite Mechanical Properties:

i) The current knowledge of the mechanical response of graphite to varying environmental conditions is quite limited. To provide the required equipment and tooling to conduct future programs aimed at increasing the available information on the mechanical behavior of graphite, several pieces of equipment are either being built or in the preliminary design stage.

ii) The design of a high temperature flexure and graphite furnace assembly capable of test temperatures to 3000°C and specimen capacity up to 1 inch square is being completed. Fabrication of this unit will be completed and it will then be utilized in future flexure testing programs. Modification of the presently available graphite tensile furnace for high temperature compression and miniature flexure testing is in progress. The final assembly of a high-temperature drop weight type impact unit is in progress and will be evaluated. This unit will be used to evaluate the response of various grades of graphite to impact at temperatures up to 3000°C. A set of air bearings is currently being designed to improve the alignment of tensile specimens. These bearings will improve the alignment by the reduction or elimination of friction forces encountered with universal joints, gimbals, etc.

iii) Target dates for equipment fabrication or modification are as follows:

High temperature flexure equipment designed	July 30, 1963
Equipment fabrication completed	Sept. 30, 1963
Modification of existing high temperature graphite chamber	Aug. 30, 1963
Impact apparatus completed	Aug. 30, 1963
Fabrication of air bearings completed	Sept. 30, 1963

e) Moisture Absorption and Adsorption of Graphite:

i) The purpose of this preliminary study is to determine the amount of water pick-up at various temperatures and humidities as a function of time. Since water has a moderating effect, it is important to keep the overall amount of

water in NERVA reactors to a minimum. Also, it has been suggested that moisture-laden graphite may expand rapidly or explode on rapid heating. The information generated by this initial study will be helpful in predicting the control necessary during reactor assembly and the influence of water upon operational characteristics of the NRX Reactor.

ii) Samples of graphite will be exposed to varying humidities at various temperatures for given periods of time. This work will be completed September 30, 1963. Recommendations for further study will be made at that time if the preliminary data so indicates.

f) Evaluation of a Method for Repairing Graphite:

i) A suitable method of repairing graphite components will be studied. Some inner reflector cylinders have been observed to have internal flaws. There is a real possibility that flaw-free material in the required size cannot be procured in the near future. If the cylinders are to be structurally sound, the effect of an irregular flaw may be greatly reduced by a careful repair.

ii) A method consisting of coring out the defect and bonding of a repair plug with a very thin carbon cement bond will be evaluated using extruded H4LM graphite. All of the variables (tolerance of bond thickness, type of cement, curing cycle and plug design) will be sufficiently explored to prepare an adequate specification.

iii) Evaluation of this method will be started by August 1, 1963.

4) Tie Rods:

a) The core tie rods are among the most critical structural components in the NERVA reactor; hence, it is of vital importance to establish their reliability. A fabrication development program aimed at developing a suitable process for making tie rods has just been completed, and the primary task remaining is to evaluate the resulting as-fabricated and heat-treated tie rods. The use of nonprecipitation-hardening alloys would be desirable from the standpoint of retention of high strength at high temperatures. Therefore, dispersion hardened materials are of interest.

b) As-fabricated tie rods will be examined and evaluated to determine the effects of the fabrication processes on the mechanical properties, microstructures, and soundness (freedom from macroscopic flaws, such as laps, cracks, seams, etc.). Also, the heat treatment of tie rods will be studied to determine the effects of fabrication procedures. Conceivably the "standard" heat treatment may require modification. Investigation of dispersion-hardened alloys, based on the process development by DuPont to produce T. D. Nickel, will be undertaken. See Section III.C.

c) The evaluation of the tie rod fabrication process and the resulting tie rods will be completed by August 30, 1963.

5) Titanium Forgings:

a) Recent data from the literature confirm the choice of the 5% aluminum - 2-1/2% tin - titanium alloy for the critical forged support rings at cryogenic temperatures. However, a continuation of the evaluation of production forgings using material machined from the forgings is desirable to determine anisotropy of mechanical properties and the effect of processing on microstructure. Also there is only one vendor who can make the dome end ring by a closed die approach. Thus, other possible manufacturing methods, such as spinning will be investigated. Spinning is done at a low enough temperature to result in the working of a 100% alpha grain structure which, upon recrystallization, should result in an equiaxed microstructure. Forging, on the other hand, is performed in the "alpha plus beta" field which introduces the risk of grain growth. More important, however, is that upon cooling the transformation of prior beta results in the growth of long, thin secondary alpha grains. Numerous investigations have shown that the equiaxed structure generally yields a more ductile product - hence the current interest.

b) The mechanical properties of titanium ring forgings for the core support, dome end outer reflector support and bottom support will be determined from excess stock on the forgings. Notched and unnotched tensile properties and impact properties will be obtained in the axial and tangential directions over the operating temperature ranges.

A mechanical property and microstructure evaluation of a finished, full-size, spun dome end ring will be initiated. This evaluation will entail a macrostructural examination of the cross-section, a detailed microstructure and mechanical property survey of the critical flange areas, and a general mechanical property and microstructure survey of the entire ring. The results of the evaluation will be compared to those received from the in-process study of the first, prototype, close die dome end forging.

c) Target dates:

Completion of spinning	Sept. 30, 1963
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Mechanical testing of closed-die-forged rings	Sept. 15, 1963
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6) Cluster Plate Materials:

a) The material for cluster plate application in the NERVA engine must have a high thermal neutron capture capability to suppress the thermal neutron flux peak which would otherwise cause heating problems at the top of the core. In addition, the material must have structural capability (tensile yield strength of about 10,000 psi), and be metallurgically stable in a hydrogen atmosphere or a hard vacuum in the temperature range -320 to 1000°F.

Several commercially available materials and one development alloy have been considered for cluster plate application. Among these were the commercially available hafnium, a silver-indium-cadmium alloy, and B¹⁰ bearing stainless steel; and the development alloy 304 stainless steel containing about 1% gadolinium. Hafnium has definitely been eliminated because it has no advantage over Ag-In-Cd, it hydrides, and is considerably more costly. The Ag-In-Cd alloy does not have sufficient strength at temperatures over 600°F; however, it has been evaluated and selected as an alternate for the NRX-A test series because the environments in these early tests are not severe. B¹⁰ stainless should have the high-temperature strength and metallurgical stability required. The cryogenic properties and especially the behavior of this material in the combined cryogenic and irradiation environment are of concern, however. The addition of boron to the stainless

matrix reduces the ductility of stainless significantly, and this effect has not been fully evaluated at cryogenic temperatures. The products of the neutron capture process by the boron atom are a lithium atom and a helium atom, which may impair the properties of the cluster plate. Combined cryogenic and irradiation effects have not been evaluated for this alloy. Problems in producing a B¹⁰ stainless with a high degree of homogeneity have also been encountered. B¹⁰ stainless will be evaluated as an alternate for at least the NRX-A test engines.

Because of the multitude of limitations in the above alternate cluster plate materials and of some favorable data on an experimental melt, it was decided to develop a gadolinium-bearing stainless steel. Several experimental melts made to date have been encouraging. Excluding development costs, a gadolinium stainless would be about 20% the cost of B¹⁰ stainless. To some extent in small heats and to a very large extent in large (500-1500 lb.) production heats, difficulties in hot breakdown of the cast structure have occurred. Further difficulties in recovery of gadolinium due to furnace reactions have become apparent. The need for a suitable cluster plate material, cost considerations, and the possible use of this material for other NERVA applications such as shipping wires dictates that further development effort be expended on gadolinium bearing stainless steels.

b) Evaluation of the mechanical and chemical properties of B¹⁰ stainless will be done to meet long range material objectives. To reduce costs, much of the evaluation will be on natural boron stainless, but the conclusive work will be on the B¹⁰ material. A limited amount of development work on melting and fabrication may be required depending on the results of the evaluation work and on the status of the gadolinium stainless project. Irradiation effects data will be generated on the B¹⁰ material next year if it is warranted (see III.D - GTR Irradiation No. 4).

Several approaches to the gadolinium stainless cluster plate problem will be employed. These are:

1. Preparation of the alloy by various methods of melting and subsequent fabrication into strip by forging and rolling;

2. Preparation of the alloy by pressing and sintering the metallic powders, followed by rolling into strip;
3. Preparation of strip directly by sandwiching gadolinium foil between strips of stainless steel; and
4. Depositing a coating of gadolinium directly on plain stainless steel plates by vapor deposition process.

To meet the immediate NRX-A objectives, cold worked and annealed plate of 80 Ag - 15 In - 5 Cd is being evaluated. Tensile properties will be established from 300 - 750°R for cold worked plate and from 300 - 1400°R for annealed plate. For increased strength, the tensile properties of 75 Ag - 11.5 In - 8.6 Cd - 4.4 Sn will be evaluated from 300 - 1400°R.

The product of any of the above approaches will be given thorough evaluation of mechanical and chemical properties. The best product will be examined for irradiation effects (see III.D)

c) Target dates:

304 Gd

1st air melt	July 15, 1963
1st arc melt	Sept. 1, 1963
1st compaction	Aug. 10, 1963
evaluation complete	Sept. 20, 1963

304 B¹⁰

Tensile testing of natural boron strip and bar completed	Sept. 1, 1963
Initiate testing on B ¹⁰ strip	Sept. 15, 1963

Ag-In-Cd

Ag-In-Cd Properties established	Aug. 30, 1963
Ag-In-Cd-Sn evaluation started	July 15, 1963

7) Evaluation of Aluminum Alloys:

a) Aluminum alloys constitute an extremely important class of structural materials for use in space vehicles generally, and for the NERVA reactors specifically.

Applications of high-strength alloys frequently require that they be welded, and this results in a degradation of mechanical properties which often cannot be recovered by subsequent treatment. Selection of welding method, heat treatments, and alloy must be carefully made to ensure that the desired properties are obtained in the finished structure. Introduction of a new commercially available alloy is, therefore, a matter of immediate importance. Kaiser Aluminum Company has just announced availability of a new Al-Mg-Zn alloy designated as 7039. Judging from preliminary data supplied by the manufacturer, it appears that this alloy may have significant advantages in some applications as compared to those presently specified (2219 and 6061).

b) Experimental work will be undertaken to become familiar with the desirable features and the limitations of this alloy. Bend, impact, formability and tensile tests of weldments with and without heat treatment will be performed. Resistance and fusion methods of welding will be investigated; thickness of the material will be from 0.032 to 0.500 inch; filler metal will be used in most cases, but some welds in thin material will be autogenous.

c) Target dates:

Receipt of material	July 20, 1963
First fusion weld specimens ready for testing	Aug. 15, 1963
First resistance welds ready for testing	Aug. 30, 1963

8) Beryllium Components:

a) Surface damage effects:

i) Beryllium is susceptible to surface damage induced during machining operations. During reactor operation, a damaged surface condition could lead to the formation of cracks which could propagate under the thermal

stresses imposed during reactor operation. Hence, it is necessary to prevent or counteract damaged surface conditions on beryllium component parts.

ii) The significance of surface damage will be evaluated by imposing various surface damage conditions on samples of beryllium and subsequently evaluating the mechanical properties of these specimens. Likewise, the effectiveness of various thermal and chemical treatments in eliminating surface damage will be evaluated. Evaluation will be based upon tensile tests, microscope-stage bend tests, and metallographic examination.

iii) Target dates:

Specimens machined	Aug. 30, 1963
Chemical etching effects established	Sept. 30, 1963

b) Evaluation of Material Reclaimed from Scrap:

i) Because of the large stock pile of beryllium scrap possessed by the A.E.C., future NERVA orders for beryllium components may be required to be produced from powder reclaimed from this scrap. Inasmuch as the characteristics of the reclaimed material may differ, in either pronounced or subtle ways, from those of virgin material, an evaluation is essential.

ii) This project will involve learning the characteristics of this scrap, determining the state-of-the-art in reclaiming it, searching for evaluation of components made from similar scrap in the past, planning an evaluation of components made from the scrap, evaluating the results, and determining how beryllium scrap can be used for NERVA hardware.

iii) Target dates:

Discussion of problem with A.E.C., SNPO, and industry	July 31, 1963
Proposed evaluation program	Aug. 15, 1963

9) Other Materials Problems:

Problems associated with hardware procurement and material data development are expected with other alloys considered for NERVA applications, such as 300

series stainless steel for springs, 400 series stainless steel for roller bearings, aluminum alloy for shield and core support, molybdenum alloys for cone supports, etc. Results of vibration and cold flow tests may require post-mortem materials investigation. These miscellaneous materials are not presently causing concern, but problems may arise which require action.

10) Galling and Wear of Components:

a) Titanium bolts are being used in many NERVA applications. The choice of titanium as a bolt material, although highly justified, precludes its ability to be tightened and loosened several times during assembly and then to be tightened and disassembled after testing. This requirement, therefore, necessitates work in the area of antigalling coatings for titanium, since the material cannot be repeatedly tightened, loosened, and retightened without seizing. This work is urgent because the current method, (the best now known), of preventing titanium fastener galling has not been evaluated with respect to its effect on the titanium. This method consists of silver on electroless nickel plate on a flash zinc-coated surface.

b) Work will be initiated wherein the present coating and other coatings such as silver and gold electroplating, phosphatizing, etc., will be applied to bolts and evaluated under reactor conditions. A simple procedure of tightening and loosening bolts several times followed by subjection to a cryogenic and elevated temperature hydrogen atmosphere should be adequate to evaluate the capability of the coating.

c) Target dates:

Completion of experimental plans	Aug. 15, 1963
Acquisition of test materials	Sept. 15, 1963

C. Fabrication Development

1) Control Drum Plate Fabrication Development:

a) Although the development of production processes for aluminum-boron control plates is essentially completed, some further effort will be applied to determine the mechanical properties, to reduce scrap, and to recover boron from scrap.

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b) The effects of testing speed and temperature on tensile properties will be investigated first, with tests to be run at -320°F , room temperature, and 150°F , and at strain rates of 1, 0.1, and 0.01 in/in/min. Ductility is of particular interest, and the effects of surface condition on this property may also require investigation. Tensile tests are to be followed by bending and fatigue tests.

c) Scheduled completion dates for the mechanical tests are as follows:

Tensile tests	Aug. 9, 1963
Bend tests	Aug. 23, 1963
Fatigue tests	Sept. 20, 1963

2) Joining of Graphite to Graphite and to Other Materials:

a) Processes for joining graphite to itself and to other materials are essential in modular construction of NRX reactors. Since the NERVA applications require controllable bonding and joining procedures of a reproducible nature, a planned program will be needed to derive this data.

b) Specific joining problems to be investigated include:

i) Joining H4LM graphite to pyrolytic tile with epoxy cement and carbonaceous cement.

ii) Joining 304 stainless steel sheathed thermocouples to graphite for service at cryogenic temperatures.

iii) Joining of 304 stainless orifices to graphite with a cement which meets the following requirements:

(a) Must maintain a seal under pressure differential of 70 psi at 200°F

(b) Must not clog the orifices, when subjected to 540°F . (Many cements boil and expand tremendously at these temperatures).

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In addition, work will be initiated, in preparation for C.Y.'64, on the problems of:

- iv) joining fuel elements together and to graphite transition blocks to form structural modules,
 - v) joining transition blocks to tubular graphite modules (including cloth laminates),
 - vi) joining module assemblies to metal and graphite inlet end caps, and,
 - vii) joining pyrolytic tile to metal and graphite materials (including cloth laminates).
- c) Work to be completed by September 15, 1963. (Items i - iii)
- 3) Evaluation of Colloidal Silica Impregnation of the Graphite Inner Reflector Cylinder:
- a) In certain NERVA applications, it is necessary to reduce the permeability of graphite to hydrogen. By applying an impermeable impregnation to graphite parts to be used in a hydrogen atmosphere, undesirable hydrogen leakage through graphite will be substantially reduced or eliminated.

b) A colloidal silica impregnant, which was developed by LASL will be applied to graphite test samples. However, this colloidal silica coating relies on a sodium silicate or sodium hydroxide bonding medium which is weak if all water is removed from the silica. These impregnated samples will be examined metallographically for the depth of impregnation by the colloidal silica. Also, these samples will be evaluated for effectiveness in reducing leakage of hydrogen at elevated and cryogenic temperatures or under a thermal or pressure gradients simulating NERVA applications.

An experimental impregnating apparatus has been built. It will be used in evaluation of the colloidal silica impregnation of graphite. The apparatus can be pressurized to force the colloidal silica particles into the graphite surface, can be evacuated and can be heated to moderate temperatures to drive off excess water.

c) Target date:

Experimental Impregnation Device evaluated August 15, 1963

4) NRX-B Development of Fabrication Methods for Producing Modular Shapes from Laminated Graphitic Composites:

a) A modular core construction may require a degree of toughness in structural parts which is not normally found in graphite. Recent development of laminated graphite composites (PT Grades) offers promise of producing such structural parts which would appear to have greater flexural and impact toughness.

b) A fabrication development effort will be initiated on short modular shapes of the laminated graphite composites. The characteristics of such small scale samples will be evaluated for dimensional control, ease of machining, requisite physical and mechanical properties, response to vibration, flexural bending or shock and possible limitations which would interfere with efforts to scale up the component size. This effort will run concurrently with the physical and mechanical property testing program on separately prepared blanks.

c) Initiate the fabrication development effort July, 1963.

- 5) NRX-B Development of Shaped Pyrolytic Graphite Thermal Insulation:
 - a) Pyrolytic graphite, a highly anisotropic material, is required as a thermal barrier in the cooled bottom support. Conventional methods for deposition do not permit a component to have sharp changes in contour to give thermal insulation in all the requisite directions of such a component. The development of plasma arc spray deposited pyrolytic graphite may make it possible to produce components with the necessary sharp curvature to fit design configurations.
 - b) A fabrication development effort will be initiated to make desirable hardware shapes by spraying onto properly shaped mandrels. The dimensional stability, microstructure, thermal conductivity and strength of trial components will be evaluated as a function of process variables and thermal cycling.
 - c) Initiate fabrication development effort July, 1963.
- 6) Evaluation of the Effect of Metallurgical Processes on Materials:
 - a) A program will be included during this period to evaluate the effect of such metallurgical processes as welding on three new potential structural materials, aluminum 7039, pure nickel and titanium alloy (extra low interstitial). In preparing process specifications, approving vendor specifications and applying the process to the design, the reliability, cost, and delivery depend upon a clear knowledge of the capability for any process to produce obviously (or potentially) undesirable conditions, (for example porosity, undercut, high residual stresses, etc.) and the relative undesirability of each condition thus generated in terms of actual component performance. Effective process control applies the minimum control consistent with avoiding the occurrence of any truly detrimental condition. To obtain such information existing information must be refined and, for the newer materials, supplemented.
 - b) The experimental portion of the program will consider the general effect of joining, forming, and heat treating processes on the material with regard to achieving the best combination of strength and accuracy in the finished component. Optimum in-process metallurgical conditions will be established to assure maximum ease of forming and joining. To assist in relating the results of generalized

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applications of the metallurgical processes to actual performance during manufacture or service, currently used materials such as 2219 aluminum and one nickel-base alloy will be carried through the program for comparison.

c) Material procurement:

Aluminum Alloys	July 31, 1963
Ni Base Alloys	Aug. 31, 1963
ELI Titanium	Aug. 31, 1963
Pure Nickel	July 31, 1963

7) Process Development - Joining (Brazed Instrumentation Pass-Through)

a) Although the development of the no-flux, low temperature brazing process has been completed in the previous period, some further effort will be required before the process is applied to a reactor assembly.

b) Qualification samples will be brazed in the actual production brazing rig and subjected to proof tests as well as leakage tests. Mechanical adoption of the brazing rig to the assembly fixture must be accomplished, an operator trained and a set of operating instructions prepared.

c) Target dates:

Qualification Test Pieces	July 31, 1963
Operating Instructions and Operator Training	Aug. 30, 1963

D. Irradiation Effects

1) Nickel and Iron Base Precipitation Hardening Alloys Inconel 718, Inconel X-750, Hastelloy R235 and A286.

a) The four alloys to be studied in this irradiation experiment are candidate materials for tie rods and other hardware applications in NERVA. The combined effects of cryogenic temperatures and large neutron doses compound the problems associated with the application of these materials, since radiation damage effects tend to be frozen in at low temperatures. There is a great need to generate applicable basic mechanical property data on these materials in a simulated NERVA environment.

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b) The Ground Test Reactor (GTR) in Forth Worth has been selected as the site for carrying out the planned irradiation of these materials. Approximately 100 tensile specimens will be irradiated in liquid nitrogen (-320°F). Some of these specimens will be tested at -320°F in special equipment immediately after irradiation without an intervening warmup to room temperature. Testing in this manner is important because a minimum annealing of damage is permitted to occur. Other specimens will be annealed at room and elevated temperatures to determine any deleterious effects which could be caused by coalescence of defects or recovery of properties due to removal of defects by any of several mechanisms. Pre-irradiation and post irradiation control tests will be performed in the special tensile equipment. Extensive metallography will be employed on samples which deviate from the range established by the control tests.

c) Target dates:

Fabrication of tensile specimens and special grips	July 27, 1963
Start 150 hour irradiation run	Aug. 27, 1963
Tensile tests completed	Sept. 30, 1963
Metallography completed	Oct. 31, 1963
Final report	Dec. 2, 1963

2) Preparation for 8910 Program

a) To meet milestones established for C.Y. '64, a considerable amount of work must be initiated in C.Y. '63. This work includes procurement and inspection of test materials, development of special in-pile thermal conductivity apparatus and the design and development of irradiation capsules.

b) Because of the high costs associated with irradiation experimentation, test materials must be given close quality examinations before they can be utilized. Chemical, radiographic, ultrasonic and other non-destructive tests are time consuming but necessary.

In-pile thermal conductivity experiments have not yet been attempted. While it is technically feasible to do this, a considerable development effort is required.

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The design and development of capsules to contain experiments planned for next year is not expected to pose unusual problems.

c) Target dates:

Initiate procurement of test materials for January and May GTR tests	July 8, 1963
Initiate development of in-pile thermal conductivity apparatus	July 15, 1963
Initiate fabrication of prototype irradiation capsule	Sept. 23, 1963

3) NRX-A Irradiation Effects on Instrumentation Devices:

a) Early cryogenic irradiation tests on instrumentation devices have demonstrated that materials of construction and design styles were the cause of some failures. Consultation with the suppliers of pressure transducers, accelerometers and linear motion displacement transducers has resulted in significant changes in the materials of construction and in the design style. Improved instrumentation devices are currently scheduled for irradiation testing. Other devices such as thermocouples, resistance thermometers, strain gages, neutron detectors and preamplifier components are also subjected to unusual irradiation environments and require critical analysis for optimized designs.

b) Careful post-mortem examination of irradiated instrumentation devices will be done to determine what changes have taken place and the cause of any failures encountered. A review will be made of the problem to determine possible changes in design or materials prior to further testing. This will be a continuing effort into C.Y. '64.

c) Target dates:

Examine irradiated devices from July test	August, 1963
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III. NE 8910 Program: October, 1963 - September, 1964

A. Vendor Liaison, Consultation and Evaluation

Vendor liaison, consultation and evaluation will continue to require significant effort to improve the product and the delivery and, in some cases, the cost. Although vendors of most of the materials used in NERVA will be contacted, emphasis 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 re-evaluate manufacturers and check for progress in solving present problems.
 - b) To realistically revise present specifications which are inadequate.
 - c) To help develop better products.
 - d) To examine new products which may do existing jobs better.
- 2) With all conventional graphite manufacturers:
 - a) To make an initial evaluation of their processes, facilities, and grades to solve a number of existing problems.
 - b) To realistically revise present specifications which are inadequate.
 - c) To help develop better processes and/or grades and/or larger sizes of existing grades to solve such problems as exist in obtaining flaw-free barrels.
 - d) To evaluate new grades and/or products which may do existing jobs better.

Target Dates:

Technical discussions will be held with all appropriate graphite vendors by December 30, 1963.

Technical discussions will be held with all appropriate beryllium vendors by November 30, 1963.

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B. Materials Evaluation, Application and Behavior

Studies of materials evaluation, application and behavior are continuing activities needed by design, manufacturing, quality control and assurance, and purchasing personnel. Tasks to be done include:

1) Material Data Sheets:

a) Designers need a materials manual as a source of data for design purposes.

The Material Data Sheets are intended to provide the special types of materials data needed by WANL designers, particularly the mechanical and physical properties as a function of temperature.

b) Where available, this information will be obtained from the literature or from vendors. The review of the important materials requirements of NERVA components and the priorities subsequently determined will guide the acquisition of data. Critically needed data will be obtained by experiment.

2) Consulting:

Consulting will continue to be a major effort, for numerous requests for advice 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, evaluation of suppliers, etc.

3) Graphite Mechanical Behavior:

a) Engineering Properties

i) Statistically significant mechanical and physical property data will be established for many grades of graphite and graphitic products as functions of crystallographic direction, temperature, testing atmosphere, and position within raw material. These property data are greatly needed to allow efficient evaluation of the grades and better design of reactor components (both for present designs and for future designs).

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ii) Statistically significant mechanical and physical property data will be obtained for approximately 25 to 30 grades and/or products for those environmental conditions peculiar to the NERVA system and not covered by the work of other laboratories. Tensile strength, flexural strength, compressive strength, shear strength, density, thermal expansion, thermal conductivity, modulus of elasticity, fatigue strength, ductility, and Poissons ratio will be determined as functions of the variables listed in part i). These data will be assembled and incorporated into the Materials Data Sheets.

iii) Target dates:

Room temperature tensile data and flexural data (ATJ & ZTA)	April, 1964
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Thermal expansion data (-300° to 4500° F)	April, 1964
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b) Effect of Structural Variables

i) The solution to the problem associated with the use of graphite, both binder types and pyrolytic, for structural applications in the NERVA engine requires as complete an understanding of the mechanical behavior of these materials as possible. The current status of knowledge regarding the effect of structural variations of graphite from both the macroscopic and microscopic scale is rather limited. The work to be done is intended to supplement present knowledge and in certain cases to examine variables that have received little or no attention to date. This work is required to predict the behavior of actual graphite hardware components and to develop the understanding of what design approaches would be fruitful in the design of new components and development of new graphitic materials.

ii) Several programs will be conducted; they are summarized below:
This will initially be done at ambient temperature but will later be extended to cover the temperature range from room temperature to approximately 3000° C. After a clear understanding of the role played by test volume and the effect of temperature is gained, variable strain rates will be examined for selected cases.

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This work will be done for graphite grades being used in the reactor and for any special grades under consideration for advanced designs.

- (a) The effect of stress concentrations (notches, etc.) will be examined in conventional tensile and flexure tests as a function of strain rate and temperature. The K_t of these stress concentrations will be varied over as wide a range as feasible.
- (b) A study of the deformation and fracture mechanism as a function of temperature will be conducted for both conventional extruded and molded grades and pyrolytic graphite. This will include an examination of the path and mode of fracture as well as possible plastic deformation mechanisms at the elevated temperatures. Techniques for crack initiation and propagation studies will be developed and the behavior of existing cracks studies as a function of testing temperature. This study will attempt to define the weak link in various types of graphite.
- (c) The strengthening of all types of graphite by high temperature deformation will be examined. Limited available data indicate that pyrolytic graphite can be strengthened by prior plastic deformation at elevated temperatures above the final test temperature (lower than the temperature used for initial deformation). This type of mechanical treatment if applicable to bonded grades of graphite, would offer one means of improving low-temperature strength values.
- (d) The effect of cycle frequency on the fatigue strength as a function of temperature will be determined. Since the properties of graphite are rather sensitive to strain rates, it is desirable to know how various rates of cycling affect the flexural fatigue strength of typical grades of graphite. The literature indicates that the fatigue strength versus temperature behaves in a manner similar to the tensile behavior. The effect of cyclic rate has not been examined and could have a pronounced effect on fatigue life.
- (e) Since graphite is basically a brittle material, work will be initiated to investigate the influence of surface conditions, i.e., machining finishes, machining

techniques, effects of coatings, environmental conditions, etc., on the tensile strength at room and elevated temperatures.

- (f) The tensile strength of graphite varies with the volume of the piece in a manner proportional to the probability that the volume contains a flaw of sufficient size or shape to cause failure at low stress. This volume effect will be studied to allow proper use of property data in design. The tensile strength of a number of grades of graphite will be obtained for varying volumes of uniform maximum stress. The data will be evaluated and the material constants for each grade will be determined.

iii) Target Dates:

The programs outlined are, in general, of the continuing type but the following schedule can be tentatively established:

Complete literature survey	October, 1963
Order material and initiate test programs	November, 1963

Due to special material requirements, the initiation of certain phases of various test programs will be dependent upon material delivery.

c) Study of Effects of Pre-stressing on Mechanical Properties of Graphite

i) It is desirable to proof test graphite components before insertion into a NERVA reactor at a stress sufficient to sense failure of defective ones. However, proof testing at too great a stress level will cause damage in originally sound components. Therefore, the stress level that will cause failure of defective components without harming sound components will be determined. This work will be coordinated with the fuel program work along similar lines.

ii) Graphite tensile specimens will be prestressed at stresses equal to the mean tensile strength minus 1, 2, and 3 standard deviations. This prestressing will be done 1, 2, and 5 times to study the effects of cycling. The tensile strength of these pre-stressed specimens will be compared to the tensile strength of unstressed specimens, and the safe limit of prestressing will be obtained. This will be done for several grades of graphite.

- iii) Initial data for ATJ will be obtained by January, 1964.
- d) Study of the Effects of Superimposed Stresses on Graphite
 - i) Components in the NERVA reactor endure vibratory stresses superimposed on constant stresses. At present, the effects of these superimposed stresses is unknown, but it is suspected that they are quite detrimental (i.e., that the modified Goodman Diagram is quite depressed as it is for other brittle materials such as cast iron). This effect has often been proposed as a major reason for KIWI failures. Data to be generated in this study are necessary for proper design.
 - ii) Graphite specimens, both tensile and flexural, will be subjected to varying superimposed stresses at varying frequencies. The types of superimposed stresses will include tensile to compressive, tensile to tensile, and compressive to compressive. Modified Goodman Diagrams will be made for all of the various combinations for several grades of graphite. Initial work will be done at room temperature.
 - iii) Initial testing will start in January, 1964.
- e) Study of the Effects of Coatings on the Mechanical Strength of Graphite
 - i) At present, it is known that a niobium-carbide coating increases the strength of some grades of graphite (e.g., RVC) and decreases the strength of others (e.g., ATJ). This effect is probably related to the effect of the coating on notch sensitivity. The specific effect is not known for specific grades of graphite being used in NERVA. Data on the specific effect are required for those grades being coated to allow for efficient design.
 - ii) For each grade of graphite being niobium-carbide coated, the tensile strength and flexural strength will be determined with and without the carbide coating. The effects of the coating will be determined. Geometry of coated parts, depth of coating, and temperature of testing may be important variables and will also be studied.
 - iii) Initial testing will start in February, 1964.

f) Effects of Hydrostatic Pressure on the Mechanical Properties of Graphite

i) It has been suggested that the mechanical properties of graphite may be improved by subjecting the material to a hydrostatic pressure. This effect of pressure should vary with the porosity of the graphite and with the hydrostatic fluid. If the mechanical properties are significantly improved, several NERVA reactor components may be redesigned more efficiently.

ii) Graphites of varying degree of porosity (including impregnated and coated material) will be subjected to hydrostatic pressures of up to 1000 psi during tensile testing in several atmospheres. All initial work will be done in nitrogen or argon at room temperature. If there is any effect, work will proceed at elevated temperatures. Both the inert and hydrogen atmospheres will be used at these elevated temperatures to determine if there is any difference due to chemical interactions. If there is a significant improvement in the mechanical properties, the most promising graphites will be retested in hydrogen at elevated temperatures to determine if there is still an increase in mechanical properties, and if so, over what range of conditions.

iii) Target dates:

Apparatus design completed	October, 1963
Apparatus constructed	December, 1963
Testing initiated	January, 1964

g) Effects of Hydrogen Atmosphere on High-temperature Creep of Graphite

i) The high-temperature creep rate of porous grades of graphite is greatly increased in a hydrogen atmosphere. However, neither the mechanism of the increased creep rate nor the exact increase in the creep rate is presently known. With this creep superimposed on the high temperature creep and the irradiation-induced creep, the total combined creep may become an important problem in NERVA reactors. Therefore, the increased creep rate due to a hydrogen atmosphere will be determined to help estimate the total amount of creep.

ii) Creep specimens of several grades of graphite will be pulled in a high temperature creep apparatus with both hydrogen and argon atmospheres. Data will be generated as a function of temperature and the increase in creep rate will be determined for each temperature. The temperature threshold of this phenomenon will also be determined.

iii) Target dates:

Test apparatus completed	December, 1963
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Initial testing of creep specimens	January, 1964
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h) The Effect of Flowing Hydrogen Upon Graphite Structural Components at Elevated Temperatures

i) Corrosion of graphite by hydrogen has been observed at core temperatures. A program has been initiated to study this effect on fuel elements using equipment designed specifically for fuel element testing. A parallel hydrogen corrosion study is needed for structural graphite. Some data will be needed to correlate the severity of corrosion with various geometric features, such as sharp corners, orifices, and orifice constrictions. The rate of corrosion of various types of graphite will be related to the binders and porosities.

ii) Parts of given geometrics of various types of graphite will be subjected to gaseous hydrogen at several flow rates, pressures, and temperatures to simulate core environments. The effect of methane upon the rate of hydrogen corrosion will be studied. Data will be established in terms of weight loss, changes in average pore cross section, and dimensional changes of test specimens.

iii) Target dates:

Hydrogen corrosion test apparatus completed	February, 1964
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Initiate testing	March, 1964
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4) Cluster Plate Materials:

a) Because of the large volume of work outlined in II. B 6, considerable carryover of the evaluation of B¹⁰ stainless and the development and evaluation of Gd stainless materials into CY '64 is necessary. Furthermore, the needs of NERVA are expected

to expand and new applications for poison and control materials arise. After acceptable materials are developed in time to meet hardware schedules, it will be possible to work on improving the materials and reducing their cost.

b) Completion of the mechanical and chemical property evaluation of the B¹⁰ stainless heat will be accomplished. Development of methods to reduce the problems of fabricability and homogeneity in the product and experimenting with various methods of hot breakdown to improve the properties will be initiated if necessary.

The evaluations initiated in CY '63 will be completed. The exact approach to be taken on the development of gadolinium stainless will become clearer when the results of the initial attempts are known. For example, if air induction melting is unsuccessful, vacuum arc melting will be employed. Arc melting in a water cooled copper vessel should eliminate recovery problems, but may introduce problems in methods of Gd addition. Extrusion methods will be tried to improve yield after hot breakdown. Cost reduction through production of larger heats, compacts, or batches will be undertaken.

c) Target dates:

Complete evaluation of B ¹⁰ stainless	November 15, 1963
Complete evaluation of sandwich and coating method for 304 Gd	November 15, 1963
Evaluation of arc melted material	November 1, 1963

5. Beryllium Components:

a) Surface Damage Effects

i) Beryllium is susceptible to surface damage induced during machining operations. This damaged surface, as noted previously, could lead to the formation of cracks which could propagate under the thermal stresses imposed during reactor operation. Hence, it is necessary to prevent or counteract damaged surface conditions on beryllium component parts.

ii) Surface damage effect on beryllium of various compositions (present commercial grades used in NRX compared with highest purity commercial grades) and produced by vacuum hot pressing and extrusion processes will be studied.

iii) Target dates:

High purity material received	October 1, 1963
Program completed	February 1, 1964

b) Evaluation of Material Reclaimed from Scrap

i) Because of the large stock pile of beryllium scrap possessed by the AEC, future NERVA orders for beryllium components may be required to be produced from powder reclaimed from this scrap. Inasmuch as the characteristics of the reclaimed material may differ from those of virgin material, evaluation is essential.

ii) This program, begun in CY '63, will be continued. A comparative control test program will be conducted to include tensile properties, metallographic examination, and complete chemical analysis (to predict induced radioactivity resulting from ground test operation of the NERVA reactor). This is necessary to establish whether any dissimilarities exist between the virgin and reclaimed materials. Sufficient purity might permit cold disassembly and reuse of beryllium reflectors within a month of operation.

A work schedule will be established after the August 15, 1963, program (see II. B. 8. b) has been reviewed and approved.

c) Mechanical and Physical Behavior of Beryllium

i) The mechanical and physical properties of Be of a given composition vary markedly with the process employed in making the material (e.g., hot pressing, extruding, forging, rolling, etc.). There is also considerable variation in properties of material produced by a given process. The amount of data available, particularly at the temperature extremes, is inadequate for establishing reasonable property values for design purposes.

ii) The mechanical and physical properties of Be will be evaluated as a function of composition (ranging from commercially pure structural grade Be to the highest purity Be available commercially), heat treatment, temperature (-320°F to 1200°F), and fabrication process (including hot pressing, extruding, and rolling).

iii) Target Dates:

Material available	October 1, 1963
Evaluation of properties as a function of temperature	February 1, 1964
Study on effect of heat treatment (aging and strain aging) effects initiated	March 1, 1964
Heat treatment effects completed	September, 1964

6) Titanium Forgings:

a) This project is a continuation of the evaluation of spinning (see II. B. 5) as a means of forming the difficult-to-make dome end support ring. At present, it is made by closed-die forging and only one vendor is equipped to make it by this method. Therefore, another forming method, such as spinning, is desirable.

b) A mechanical property and microstructural evaluation of a finished, full-sized, spun dome end ring will be completed. This evaluation will entail a macrostructural examination of the cross section, a detailed microstructure and mechanical property survey of the critical flange areas, and a general mechanical property and microstructure survey of the ring. The results of the evaluation will be compared to those received from the in-process study of the first, prototype, close die dome end forging.

c) Target date:

Completion of evaluation	January 1, 1964
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7) Aluminum Alloys:

a) Aluminum alloys constitute an extremely important class of structural materials for use in space vehicles generally, and for the NERVA reactors specifically. Applications of the high-strength alloys frequently require that they be welded, and this results in a degradation of mechanical properties which often cannot be recovered by subsequent treatment. Selection of welding method, heat treatments, and alloy must be carefully made to ensure that the desired properties are obtained in the finished structure. Introduction of a new commercially available alloy is, therefore, a matter of immediate importance.

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Kaiser Aluminum Company has just announced availability of a new Al-Mg-Zn alloy designated as 7039. Judging from preliminary data supplied by the manufacturer, it appears that this alloy may have significant advantages in some applications as compared to those presently specified.

b) During this period, evaluation of the new alloy 7039 will be completed. Assessment of its potential for NERVA structural applications will be based on the strength, ductility, formability, and toughness exhibited by as-welded and by heat treated weldments at cryogenic, ambient, and elevated temperatures. The order of merit for various welding methods will also be established. A preliminary estimate of the utility of this alloy will be made at an intermediate stage of the investigation, and the extent of subsequent work will be dependent on the findings at that time.

c) Target dates:

Preliminary evaluation complete	January 15, 1964
Final evaluation complete	July 31, 1964

8) Dispersion Hardened Materials:

a) The search for new high-strength materials suitable for applications in the severe NERVA environments is continuous. The conventional high strength steels are not suitable because they exhibit brittle behavior below their nil ductility transition (NDT) temperatures and have limited strength at elevated temperature. The NDT temperature of a material is usually raised by neutron irradiation. Most refractory metals are unsuited for applications at cryogenic temperatures because of this NDT behavior. Most refractory metals also react with hydrogen or carbon to the extent that their structural integrity is impaired. Until recently, the only commercially available materials with properties that approached the demands of NERVA were the precipitation hardening alloys of the class which have matrixes of the austenitic, or face centered cubic crystal structure. Materials which have the fcc crystal structure do not exhibit NDT behavior. The predominant hardening mechanism of these alloys is based on the resistance to dislocation motion offered by large strain fields caused by submicroscopic,

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coherent precipitate particles. Moderately high temperatures (1250 °F) and limited fast neutron flux dosages $10^{17} - 10^{18}$ NVT are not expected to drastically affect the stability of these precipitate particles. For extended temperature and time requirements with higher neutron dosages, these materials will become marginal.

Recently, DuPont has commercialized a nickel-base alloy (TD Nickel) hardened by a dispersion of submicron particles of an insoluble second phase. This second phase is very stable to temperatures exceeding 2400 °F. Two properties of TD Nickel would make it ideal for at least one component in the NRX-B design (shroud tubes for the base plate). These are high thermal conductivity (600 BTU/ft.²/hr./°F/in. compared to 390 for nickel - 200) and high temperature capacity (TD Nickel has a 10,000 psi short time Y.S. at 2400 °F compared to the same strength for Nickel - 200 at 1200 °F). Modifications to this material, which would increase its strength, or development of new, stronger materials of this class could change many material selection decisions for NRX-A. The high-temperature capabilities of this material, plus the very good possibility of improved resistance to radiation damage, warrant a thorough evaluation of TD Nickel and suggest that some development effort be expended. Cryogenic data, and notch sensitivity and thermal properties as a function of temperature are also lacking.

b) A thorough evaluation of physical and mechanical properties of TD Nickel will be initiated and completed during CY '64. Careful examination of methods of brazing this material to itself and to pure nickel will be made to determine its applicability for base plate components. Close follow of DuPont's development program will be made and some experiments performed to determine if an improved material could be developed for tie rod applications.

c) Target dates:

Complete tensile tests on bar and
extruded shapes

March, 1964

C. Fabrication Development

1) Joining of Graphite to Itself and Other Materials:

a) An extensive program is needed to develop methods, materials, and processes for joining graphite of all types to graphite, metals, and ceramics to meet present and future design requirements. Since graphite is to be used from cryogenic temperatures to reactor core temperatures, an unusually large number of joining materials will be needed to accommodate NERVA applications for both component and core tests.

b) i) For the temperature range of liquid hydrogen temperatures to 500 °F, plastics, resins, epoxies, and synthetic rubber joint materials will be evaluated.

ii) For the same temperature range, various carbonaceous cements will be evaluated.

iii) Diffused metal and metal filler materials will be used to join graphite to other materials for service at temperatures in excess of 500 °F.

The forementioned joining materials will be used with types of joints such as lap joints, butt joints, scarf joints and with modifications or combination of all three types of joints. Evaluation of design of types of joints will be made for graphite joining applications. Analysis will be made of the optimum joint clearance in each type of materials being joined to allow for processing variables. In many cases, development will be necessary to compensate for gross mismatching of coefficient of expansion of materials being joined. Both shear and tensile tests will be performed on each joint being tested. In some cases, impact strength, torsional fatigue, and flexural bending fatigue tests will be performed. Consideration will be made for radiation damage effects and the literature will be reviewed to avoid the investigation of materials already demonstrated to be inadequate.

c) Target dates:

Graphite material procurement completed	October, 1963
Initial specimens for test	September, 1963

2) Impregnation of Graphite:

- a) The aqueous colloidal silica impregnating process has several limitations.

The water used in this process presents a major problem, since all of it must be removed from the impregnant and the graphite reflector barrel. This process is a substantial source of water, which serves as an efficient moderator and affects the operation of the reactor. Also, this impregnant is chemically buffered with sodium hydroxide, which serves a dual purpose. The hydroxide keeps the colloidal particles in suspension and effects bonding during curing of the impregnant by forming sodium silicate. When the colloidal silica coating loses water from the sodium silicate, the mechanical strength of the impregnant is reduced substantially. The impregnated materials are subjected to irradiation, hydrogen gas and thermal differentials of about 50 - 500 °R.

- b) The development of improved impregnating materials will be investigated.

Primarily, the program will be directed toward an impregnating process which does not rely upon water and alkali metal silicate buffers. One possible impregnating material would be organic silicones. These materials can be cracked thermally into silicon dioxide, carbon dioxide, and water. Alternate solutions to the problem such as coatings or other impregnating materials will be examined.

- c) Initial investigation of alternative materials and feasibility study of these materials will be complete by August, 1964.

3) Hot Forming of Pyrolytic Graphite:

a) Presently, pyrolytic graphite material for insulating tile is deposited in large sheets which deform upon furnace cool-down due to residual stresses which do not anneal. Flat tile can be machined only from this somewhat curved plate by cutting across many basal planes, thus undesirably increasing the across-grain thermal conductivity. Hot forming to flatten the curved plate would present a method for greatly decreasing the number of basal planes cut during machining.

b) Pyrolytic graphite plate stock will be hot formed at varying temperature, pressure, and time to determine the optimum process for obtaining flat plate. Micro-structural examinations of the formed material will be made to determine the effects of

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hot forming on the degree of graphitization, change in microstructure, thermal conductivity, etc.

c) Initial forming will be started in November, 1963.

4) Fabrication of Laminated Modular shapes for NRX-B:

a) A modular core construction may require a degree of toughness in structural parts which is not normally found in graphite. Recent development of laminated graphite composites (PT Grades) offers promise of producing such structural parts which would appear to have greater flexural and impact toughness.

b) The fabrication development effort and small-size component evaluation study will be continued. A review of the results and the material characteristics will be carried out to determine the logic of scaling up the tooling and process method to produce full scale components. If appropriate, the resulting full scale components will be evaluated in as complete an environmental condition as it is possible to simulate. Examination of components tested will be done to determine changes in structural integrity and properties.

c) Target date:

Establish feasibility for utilization

December, 1963

5) Pyrolytic Graphite Shapes for NRX-B:

a) Pyrolytic graphite, a highly anisotropic material, is required as a thermal barrier in the cooled bottom support. Conventional methods for deposition do not permit a component to have sharp changes in contour to give thermal insulation in all the requisite directions of such a component. The development of plasma arc spray deposited pyrolytic graphite may make it possible to produce components with the necessary sharp curvature to fit design configuration.

b) The fabrication development effort will be continued until the usefulness of the method is either established or proven unsatisfactory. If satisfactory, sample components of various configurations will be procured and tested in the hot flow test. Post mortems will be performed to evaluate changes which may take place.

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c) Target date:

Establish feasibility for utilization

November, 1963

6) Coated Graphite Components for NRX-B:

a) The design utilizing the cooled bottom support requires that hot hydrogen pass through the exhaust tubes of the plate. Pyrolytic graphite is used as a thermal barrier to protect the structural metals. It is believed that pyrolytic graphite will corrode in the hot hydrogen stream and will, therefore, need to be protected by either the direct application of a coating or by using a thin internal graphite sleeve which is niobium carbide coated. A difference in thermal expansion characteristics of such a coating and the base material may be the direct cause of spalling and catastrophic failure. Selection of materials with compatible expansion properties should greatly increase the reliability of these tubes for multiple-cycle operation.

b) Samples of niobium carbide have been produced for measuring thermal expansion over the temperature range of interest. The structural stability of the samples will be evaluated from the expansion characteristics and an adjusted coefficient of thermal expansion will be calculated if necessary. The results of the expansion study on niobium carbide will be compared with the expansion characteristics of pyrolytic graphite and other apparently satisfactory grades of graphite for use as sleeves. Coating experiments will be initiated and samples will be evaluated for microstructure, cracking, adherence, and response to thermal cycling. Samples will also be produced for hot flow test and evaluated for durability.

c) Target dates:

Review expansion characteristics,
select materials for coating

October, 1963

Initiate effort on production of coated
components

November, 1963

7) Fabrication of Two-Pass Tie Rods:

a) The design of a two-pass tie rod requires a means of fabricating a concentric set of passages; meeting rigid heat transfer requirements regarding wall thicknesses, finish etc; and involving a complex metering arrangement in the head of the bolt.

b) Composite extrusions and upset combined with chemical or electrodischarge machining will be given primary consideration in order to produce, if possible, a monolithic structure. Maintenance of proper metallurgical properties, material soundness and acceptable geometry must be assured through application of a systematic experimental program. Where possible operations will be carried out by potential vendors in order to determine and access practical process variations and, additionally supply information or costs and manufacturing feasibility.

c) Target dates:

Test specimen sketches and testing program complete	October, 1963
Order material	October, 1963
Select Vendor	November, 1963

8) Effect of Metallurgical Processes on Materials:

a) This is a continuation of a program aimed at achieving optimum process reliability, cost and delivery through effective process control on drawings, in specification and in vendor plants. Three relatively new structural materials are involved - 7038 aluminum alloy; pure nickel, ELI titanium alloy. Aluminum alloy 2219 and nickel base alloy 718 are carried to permit correlation with manufacturing and service experience.

b) A series of single pass and multipass fusion welds will be made in appropriate material thicknesses by conventional arc processes. The properties of the welds after various simulated manufacturing and heat treating cycles will be determined by welding processes. Soundness will be carefully evaluated nondestructively and confirmed metallographically. The shielding requirements for ELI titanium will be established. The process parameters required to meet MIL-W-6858 will be established for all

materials except titanium and weld performance evaluated. The metallurgical response to brazing alloys will be determined for pure nickel along with optimum joint configurations. Response to forming operations, optimum in-process conditions and dimensional stability through subsequent thermal treatments will be evaluated for all materials.

c) Target dates:

Test Program and specimens designed	December, 1963
Specimens complete	March, 1964
Testing Complete	July, 1964
Final Report	August, 1964
Intrim Report	March, 1964

D. Irradiation Effects

1) GTR Irradiation No. 3:

Structural Materials (200 hr. irradiation test):

TZM Molybdenum (bar)
5 Al, 2.5 Sn-Titanium ELI (bar)
302 Stainless Steel (spring temper wire)
310 Stainless Steel (spring temper wire)
Inconel 718 (sheet)
Inconel 718 (TIG welded sheet)
2219-T31 Aluminum (sheet)
2219-T31 Aluminum (TIG welded sheet)
2219-T852 Aluminum (forging)
Ni-Span-C 902 (bar)

a) The above materials are prime candidates for components having critical structural application in the NERVA engine. All these materials would have application outside the central core region and, with the exception of TZM, would be expected to operate in a cryogenic environment at some stage of the NERVA engine cycle. There are no applicable cryogenic irradiation effects data on the materials listed. The purpose of this irradiation study is to perform a preliminary evaluation of the tensile and

notched tensile properties of the listed materials in a simulated NRX test environment. The integrated fluxes attained at GTR in 200 hours are expected to be representative of early NRX-A and NRX-B reactor tests.

b) As in the experiment planned for August, 1963, the cryogenic (liquid N₂) irradiation of specimens will be followed immediately by in situ tensile testing of about 1/3 of the specimens. Other specimens will be annealed at room and elevated temperatures prior to testing. The annealing treatments will be useful in evaluating the effects of the decay heating cycle which follows shutdown of the NERVA engine.

Due to the variability of results historically experienced in irradiation testing, it is desirable to eliminate unnecessary materials variables when qualifying materials for NERVA application. For this reason, it has been decided to do the evaluation on the specific forms of the materials that are candidates for parts whenever possible. This approach, of course, creates new problems in gripping, fixturing, and specimen design. Although some of these problems have already been solved, there are many yet to solve. For example, the design of sheet specimens and grips have been worked out fairly well, but the design of wire specimens and grips are not. All materials to be used in irradiation experiments will be thoroughly characterized before utilization.

c) Target dates:

Specimen and grip design worked out	October 7, 1963
Specimens, grips, and special equipment fabricated	December 2, 1963
Irradiation	January, 1964
Post-irradiation tests completed	March, 1964
Final report	May, 1964

2) GTR Irradiation No. 4:

Structural and Other Materials (200 hr. irradiation test):

- 304 + B¹⁰ Stainless Steel (strip)
- 304 + Gd Stainless Steel (strip)
- Inconel 718 (brazed sheet lap joints)

Nickel - Inconel 718 (brazed lap joints)

Other materials to be selected

a) The above materials are candidates for components having structural application in the NERVA engine. Since there are no existing basic data on effects of cryogenic irradiation on the materials to be included, this experiment is necessary to generate sufficient preliminary information to guide future reactor designs and future irradiation programs.

b) The experimental approach to this irradiation will be similar to the previous GTR irradiations to the alloys. In the case of graphite materials, however, a different approach will be necessary because :

- (1) mechanical property (especially tensile) data are highly dependent on specimen size and configuration, and
- (2) there is apparently an inherently large scatter in mechanical properties which would tend to mask the effect of irradiation in small populations of samples.

c) Target dates:

Final selection of test materials	December, 1963
Complete procurement action on test materials	February, 1964
Fabrication of test specimens and special equipment	April, 1964
Irradiation	June, 1964
Post-irradiation tests completed	August, 1964
Final report	October, 1964

3) Precipitation Hardened Materials Series (PBR):

a) While total accumulated dosages approaching those of early NERVA test engine components can be achieved at the GTR, detailed studies at GTR will not be undertaken because of the low fluxes available there. A high flux reactor such as the Plum Brook Reactor offers at least three advantages:

- (1) Rate dependence can be evaluated,
- (2) simultaneous annealing effects are minimized, and
- (3) much higher dosages are practical.

Nickel base precipitation hardening alloys have been selected for detailed study because they are strong over a fairly wide temperature range, and do not exhibit brittle characteristics, even at cryogenic temperatures, since the matrix phase of these materials has the face-centered cubic structure. Several members of this alloy class have been selected for various applications in NERVA including NRX-A tie rods. The tie rods located in the central core regions are exposed to the maximum fast neutron flux in the reactor. Even though the temperature range expected is $200^{\circ} - 1200^{\circ}\text{R}$, some tie rods may accidentally be exposed to the highest temperatures of any non-graphite material should hot spots develop in the core due to the pyrolytic graphite insulation breakdown, or coolant channels blocking.

- b) The properties of these materials to be investigated are as follows:

Tensile
Thermal expansion
Thermal conductivity
Electrical resistivity (in-pile)
Microhardness

Studies of structural changes by metallographic techniques will be undertaken. Temperature of irradiation, test temperature, and flux level are the principle variables to be considered. At least three irradiations will be performed.

- c) Target dates:

Fabrication and checkout of prototype irradiation capsule	October, 1963
Fabrication and inspection of test specimens for first irradiation	December, 1963
First irradiation	January, 1964
Second irradiation	March, 1964

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Third irradiation	May, 1964
Complete all tests	July, 1964
Final report	September, 1964

4) Pyrolytic Graphite Thermal Conductivity Series (PBR):

Several grades of P. G. to be selected.

a) Pyrolytic graphite has been selected for several applications in NERVA based on its unusually good thermal insulation characteristics in the "C" direction. In one of these applications, insulation for tie rods, P. G. tubes are exposed to the most extreme conditions (for non-fuel materials) of high temperature, high temperature gradient, and high neutron flux. Since the structural integrity of the reactor depends very much on insulating the tie rods from high temperature exposure, it is necessary to accurately determine the behavior of P. G. in this extreme environment.

b) It is desired to measure the thermal conductivity of P. G. in-pile as a function of neutron dose and temperature. The problems associated with an in-pile thermal conductivity measurement are expected to be quite large. Capsule design and development is also expected to be difficult since a 5000 °R irradiation temperature is necessary. Pre-post micro and macro examinations for structural changes will be employed. Two irradiations (two capsules) are planned.

c) Target dates:

Develop necessary equipment for in-pile thermal conductivity measurements	February, 1964
First P. G. irradiation	June, 1964
Second P. G. irradiation	August, 1964
Final report	October, 1964

5) Irradiation Effects on Instrumentation Devices (Support to Instrumentation Groups):

a) Early cryogenic irradiation tests on instrumentation devices have demonstrated that materials of construction and design styles were the cause of some failures. Changes of some materials and designs have been made. Improved devices were subsequently ordered for testing, but because of inadequate available information, many "best guess"

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decisions were made on these improved designs. Joining of dissimilar materials and highly reliable electrical insulation are recognized as particular problems which may impair the reliability of the devices.

b) Careful post-mortem examination will continue in CY '64 on irradiated instrumentation devices to determine the cause of failures or changes. The problems will be reviewed periodically to determine what further work is required. In addition, a literature survey will be conducted to determine the known cryogenic irradiation effects on reliability of soldered, brazed, and welded joints in electrical conductors and structural parts. The materials of interest are aluminum, copper, brass, nickel, Inconel, certain alloy steels and selected ceramics. The literature search will also include a review of the known cryogenic irradiation effects observed on degradation of electrical insulating materials and also on their mechanical durability in a vibration or shock environment during cryogenic irradiation.

c) Target date:

Complete the literature survey

January, 1964

6) Post-Mortem Examination:

a) Examination will be required after vibration, cold flow, and hot tests on full reactor assemblies to determine the performance or extent of damage of individual components. Such information is necessary to determine which materials or component designs are inadequate for the environmental conditions encountered.

b) All major support members and all materials with known brittle behavior will be carefully examined. This will include tie rods, support rings, core support plate, bottom support blocks, and lateral support members. Materials which, because of their characteristics, will receive examination will include molybdenum, graphite, beryllium, bearing steels, work-hardened spring alloys, load carrying weld joints and Al₂O₃ control materials. The methods of examination will include dimensional stability, visual inspection, penetrant inspection and x-ray radiography when possible as nondestructive testing. Components exhibiting cracks will be sectioned for metallographic examination, x-ray diffraction, certain chemical analyses, and sub-size test specimens will

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be taken from certain components for mechanical testing. As far as possible, undamaged metal components from the vibration and cold flow tests will not be destructively examined. Examination after the hot tests will be as outlined above.

E. Specifications

1) New Materials and Process Specifications:

Preparation of new materials and process specifications will continue to require a high level of effort.

2) Vendor Acceptance of Specifications:

a) Normally, supplier negotiation would precede issuance of the specifications, but the demands of production schedules have required them prior to supplier acceptance.

b) New or revised specifications will be submitted while they are being processed. Existing specifications will be submitted in groups according to the type of material covered.

c) All existing materials specifications will have been submitted to suppliers by June 30, 1964.

3) Revisions to Existing Specifications:

a) More emphasis than in the past will be required to improve existing specifications. Supplier and customer comments, experience with core hardware, materials development data, and changing design requirements will form the basis for these revisions.

b) Specifications will be revised as the individual need arises; however, where data for one specification is applicable to several specifications, they will all be revised at the same time.

4) Design Review and Drawing Sign-Off:

Present trends indicate that design reviews to uncover potential problems and drawing sign-offs to approve choice of materials and processes will require a significant effort from the materials and processes standpoint.

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5) Equipment Specifications:

a) Specifications are needed for equipment used on the NERVA project, such as instrumentation devices, test equipment, ground support equipment, etc.

b) Equipment Specifications will be written and processed in the same manner as material and process specifications. Existing Equipment Specifications will be reviewed and revised, as required, to establish a standard format.

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<u>Program Item</u>	<u>Assigned E & S</u>
III. A. Vendor Liaison, Consultation and Evaluation	2.0
B. 1 Materials Data Sheets	0.5
B. 2 Consulting	2.0
B. 3 Graphite Mechanical Behavior	3.0
B. 4 Cluster Plate Material	0.5
B. 5 Beryllium Components	0.5
B. 6 Titanium Forgings	0.5
B. 7 Aluminum Alloys	0.5
B. 8 Dispersion Hardened Materials	0.5
C. 1 Joining of Graphite	1.0
C. 2 Graphite Fabrication to C. 6	1.0
C. 7 Two Pass Tie Rods	0.5
C. 8 Effect of Metallurgical Processes and Materials	1.5
D. Irradiation Effects	2.0
E. Specifications	5.0
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Total	21.0

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