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OAK RIDGE NATIONAL LABORATORY

MARTIN MARIETTA

Multimegawatt Space Reactor Materials Technology Assessment and Status Report for FY 1986

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OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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MULTIMEGAWATT SPACE REACTOR MATERIALS TECHNOLOGY ASSESSMENT AND STATUS REPORT FOR FY 1986

Prepared November 1986

Compiled by R. H. Cooper

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
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1.0 SUMMARY

The objective of this task is to review the material requirements for the various multimegawatt (MMW) concepts, identify the candidate materials that could be considered for these applications, and assess the technology status of these materials. As a result of this review, each of the proposed operating modes of MMW power systems appear challenging for the structural materials. In the station-keeping mode, structural and control materials will be expected to operate for 61,000 h or more at temperatures to 1600 K. In the alert mode, materials must tolerate operating temperatures to 1850 K for several days. In the burst mode, materials will be expected to operate at temperatures to 2000 K for times to 0.5 h. For all three applications, materials must demonstrate effective load-carrying capability, tolerance to neutron effects, and compatibility with candidate coolants and working fluids.

On the basis of this assessment, no engineering materials currently considered qualified for high-temperature nuclear applications were found to be developed sufficiently to assure meeting the objectives of the MMW program. To meet these objectives, numerous innovative and evolutionary candidate materials have been recommended by concept designers and material specialists. Many of these materials offer the potential to reduce MMW system mass significantly and to increase system lifetime. In general these recommendations can be organized into five generic materials groups: (1) refractory alloys, (2) composites, (3) monolithic ceramics, (4) control materials, and (5) coatings.

On the assumption that Nb-base alloys are being effectively evaluated by the SP-100 Project, the principle focus of the MMW Program in the area of refractory alloys is in Mo-, W-, and Ta-base alloys, Re and Re-base alloys, and CVD-W materials. On the basis of available creep property information for candidate alloys, the Ta alloy ASTAR-811C was found to have the largest creep property data base. Analysis of this data base suggests that the creep strength of ASTAR-811C exceeds that of all other candidate alloys at temperatures to 1600 K. Innovative alloys based on tungsten and molybdenum have been identified that hold the promise of performance greater than that offered by the Ta-base alloys; however, significant effort will be required to develop the engineering data base needed to demonstrate the feasibility of these alloys for MMW applications.

Metallic, ceramic, and carbon/carbon composites also have been identified as attractive candidates for MMW applications. The metal-fiber/metal-matrix composites offer significant advantages for high-temperature performance over conventional alloys and monolithic ceramics; however, much work remains to be performed to demonstrate the feasibility of this innovative material concept.

In the area of ceramic composites, significant progress is being made in the development of this class of material for automotive turbine applications. Additional work is required to assess the feasibility of fabricating ceramic composites to the sizes required for MMW needs, the adequacy of the high-temperature mechanical properties of these materials, and the compatibility of these materials with the candidate coolants and working fluids.

As a result of DARPA and NASA funded programs, carbon/carbon composites appear to be attractive candidates for high-temperature turbine applications and for radiator concepts. In order to realize the anticipated advantages of these materials for these applications, significant work in demonstrating the fabricability of carbon/carbon composites for high-temperature rotating applications and the development of specialized coatings for protection from the aggressive environments expected in MMW systems will be required.

MMW concept developers have identified the use of monolithic ceramics for in-core and nonreactor structural applications. For several applications, the requirements can only be met by ceramic materials. Although significant work is being funded by the DOE automotive turbine program on the development of SiC and Si_3N_4, additional research and development is required to demonstrate the fabricability and the compatibility of these materials for MMW applications.

In addition to SiC and Si_3N_4, other innovative ceramic materials have been identified by concept developers for in-core and nonreactor applications. Significant development work should be initiated soon to assess the feasibility of these materials.

Developmental and innovative coatings methods will be required to meet the diverse materials challenges of the proposed MMW reactor concepts. These specific challenges are in the areas of high-emissivity coatings for radiators, coatings to reduce wear between moving components operating at high temperatures, methods to improve the electron work function of candidate thermionic emitters, and thermal barriers to reduce temperature across component interfaces. Resolution of these important materials concerns was not completed in the previous space power era and remains to be completed by the SP-100 Project and/or the MMW Program.

Reactivity control materials (reflectors and absorbers) with the neutronic worth and ability to withstand the long-term irradiation and thermal shock anticipated for MMW concepts are not currently available. The current state of the art for control materials that have a relevance to the MMW program is embodied in the LMFBR, HTGR, and SNAP reactor programs. Control materials evaluated by those respective programs hold promise for meeting the MMW program requirements. However, the ability of these materials to tolerate the thermal shock associated with bimodal operation, to remain chemically stable in the proposed operating environments, and to resist damage during high-flux high-temperature neutron irradiation remains a large uncertainty.

2.0 INTRODUCTION

Anticipated operational requirements for the proposed MMW concepts call for possible operation in the station-keeping, alert, and burst modes. At present each mode appears to challenge the performance capability of the candidate materials. During station-keeping operation, structural and control materials will be required to withstand continuous long-term operation for 61,000 h or longer at operating temperatures ranging from 1100 K for radiators to 1600 K for in-core components. The alert mode may require materials to operate at temperatures to 1850 K for several days. Temperatures as high as 2000 K for times to 0.5 h may be required during the burst mode. The transition from the station-keeping to alert to burst mode may occur within a few seconds or less. In the range of operating temperatures for the three modes, materials used for fuel cladding, reactor structural, and/or reactor control applications must demonstrate (1) effective load-carrying capability; (2) tolerance to neutron irradiation at a fluence as high as 1.5×10^{23} n/cm^2; (3) tolerance to the thermal stresses caused by the transition from station-keeping to alert to burst mode operation; (4) compatibility with candidate reactor fuels; (5) compatibility with candidate coolants and working fluids, which include alkali metals and inert gases; and (6) fabricability into needed product forms and required component configurations.

Nuclear materials that are considered fully qualified and "technology ready" for concepts requiring immediate materials decisions are stainless steels and superalloys. The state of the art for these candidate materials is based on the LMFBR and HTGR programs. The LMFBR fuel system utilizing stainless steel cladding and UO_2 fuel has demonstrated successful long-term operation at temperatures to 900 K while in contact with a sodium coolant. The HTGR reactor program has demonstrated the effectiveness of graphite as a support material for carbide particle fuel. Advanced iron-base superalloys (e.g., Incoloy 800H) have demonstrated compatibility with the HTGR helium coolant at temperatures approaching 1100 K. Neither the LMFBR nor the HTGR programs provide the material technology needed to meet the MMW program requirements.

Extensive refractory alloy development occurred in the 1960s and early 1970s as part of the System for Nuclear Auxiliary Power (SNAP) program. Several Nb-, Ta-, W-, and Mo-base alloys were considered as candidates for SNAP program applications. As a result of SNAP program development activities, several Nb- and Ta-base alloys were shown to be compatible with UO_2 and UN fuels in 10,000-h irradiation tests at 1265 K and out-of-reactor fuel/cladding/coolant compatibility tests in flowing lithium at 1300 K. The Ta-base alloys were shown to be compatible with flowing alkali metals in tests to 10,000 h at 1470 K and 3000 h at 1640 K. These development efforts were terminated in the early 1970s, and no prototypical alkali-metal-cooled reactors having ceramic fuel clad with refractory alloys were built or operated. Many of the refractory alloys developed for the SNAP program are considered as potential candidates for selected MMW applications. However, they cannot now be considered state-of-the-art materials, and significant development efforts will be required to qualify them for MMW applications.

Successful completion of the Ground Engineering Systems phase of the SP-100 Program will build on the SNAP program technology by demonstrating the successful use of a Nb-base structural alloy in combination with ceramic UN fuel and liquid metal coolant (lithium). However, the SP-100 Program operational objective of seven years of continuous operation at a coolant temperature of 1350 K will not provide the materials technology needed for proposed MMW concepts.

On the basis of the above information, no engineering materials are developed sufficiently at this time to assure their successful use in a MMW concept that will meet current program performance goals. Further, the application of selected materials offers the potential to reduce MMW system mass significantly and to increase system lifetime. The long-term purpose of this task is to bring together the necessary national resources in the form of experienced personnel, specialized and unique facilities, and materials information to address this important technical concern. In the near term, the objectives of this task are to assess the material requirements for the various MMW concepts and identify the candidate materials that could be considered for these applications.

3.0 TASK DESCRIPTION

As indicated above, the objectives of this task are to (1) assess the material requirements for the various MMW concepts and (2) identify the candidate materials that could be considered for these applications and indicate those that may have the greatest potential. To meet these objectives requires (1) determination of the primary material functions and performance requirements for the numerous MMW concepts currently being considered, (2) identification of the candidate materials that have been recommended by system designers and materials specialists, and (3) assessment of the technical status of each candidate material.

On the basis of a review of the current MMW concepts five generic material applications were identified: (1) fuel cladding and support; (2) reactor structural including internal core support and pressure vessels; (3) nonreactor structural including piping and ducting, turbine housing and rotors, alternator housing, pumps, valves, and radiators; (4) absorber and reflectivity control; and (5) coatings for improved radiator efficiency, reduced wear between moving high-temperature components, and enhanced thermionic work functions. The primary material requirements for these functional applications are summarized in Table 1. To meet the operational requirements for the five generic material applications, the following material system should be considered: refractory alloys; composites (metal-fiber/metal-matrix, ceramic, and carbon/carbon); monolithic ceramics; control materials; and advanced coatings (see Table 2). For each of these materials systems, the following specific materials technology concerns must be addressed: (1) fabricability into required component configuration, (2) compatibility with demanding reactor and power conversion environments, (3) tolerance to radiation under prototypical reactor operating conditions, and (4) adequacy of mechanical and physical properties to meet MMW system requirements.

The information that follows provides an inventory of candidate materials that have been suggested by either concept developers or materials specialists for MMW concept applications. This information also provides a brief assessment of the technology status of these candidate materials.

Table 1. Summary of Material Requirements by Functional Area

FUEL CLADDING MATERIALS

- Tolerate operating temperatures from 1400 to 1850 K for seven years
- Irradiation tolerance to a goal fluence as high as 1.5 x 10^{23} n/cm^2
- Accommodate 1 to 3% diametral expansion without failure
- Tolerate burst power thermal stresses
- Compatible with candidate fuels, coolant, and fuel/coolant combinations
- Capable of being fabricated into product forms and required fuel system configuration (joining)

IN-REACTOR STRUCTURAL MATERIALS

Alkali-Metal-Cooled Systems

- Tolerate steady-state operation at temperatures from 1350 to 1800 K for seven years
- Irradiation tolerance to a total goal fluence of 5 x 10^{22} n/cm^2
- Accommodate up to 1% total deformation under anticipated operating conditions
- Tolerate burst power thermal stresses
- Fabricable into required product forms and components (joining)
- Compatible with alkali metals; low creep rate at temperatures to 1650 K (Rankine cycle application)

Particle-Bed Cores

- Inner frit must accommodate a coolant velocity of 0.3 m/s for seven years at an operating temperature of 1000 K and pressure of 7 MPa
- Inner frit must be compatible with He/Xe coolant, ZrC (fuel coating), and brief exposures to H_2
- Outer frit must have permeability sufficient to maintain a coolant velocity of He/Xe and H_2 of 0.24 and 13.7 m/s respectively
- Outer frit must have long-term compatibility with He/Xe and moderator material at operating temperatures to 1500 K
- Outer frit must tolerate intermittent exposure to H_2 at 2000 K

Table 1. Continued

Particle-Bed Cores (cont.)

- o Irradiation tolerance to a goal fluence as high as 1.5 x 10^{23} n/cm^2
- o Compatibility with candidate coolants and/or working fluids
- o Compatibility with fuel form for particle-bed concept

Gas-Cooled Systems

- o Compatibility of candidate coolants and/or working fluids with pressure vessel and piping materials
- o Fracture-resistant ceramics

NONREACTOR STRUCTURAL MATERIALS

Heat Transport Subsystem

- o Tolerate operating temperatures from 1350 to 1600 K for seven years
- o Compatible with candidate coolants and working fluids
- o Fabricable into required product forms and components

Power Conversion Subsystem

- o Tolerate operating temperatures from 1350 to 1600 K
- o Compatible with candidate working fluids
- o Turbine materials resistant to erosion from working fluids at anticipated operating conditions
- o Creep deformation of less than 1% at proposed operating conditions
- o High strength-to-density ratio

CONTROL MATERIALS

- o Operate for seven years at temperatures to 1600 K
- o Compatible with candidate coolants, working fluid, and claddings
- o Tolerate the thermal stresses associated with burst operation
- o Tolerate high neutron fluence

Table 1. Continued

COATINGS MATERIALS

- o Stable on candidate radiator materials having emissivity values of 0.8 to 0.95
- o Compatible with candidate materials for times to 61,000 h and temperatures to 1200 K
- o Remain intact through thermal cycles resulting from normal operation and hostile threats

Table 2. Summary of the Relationship of MMW Functional Areas and Specific Materials Technologies

Materials Technologies	MMW Functional Areas				
	Structural			Absorber and reactivity control	Coatings
	Fuel cladding	Reactor	Non-reactor		
Refractory alloys	X	X	X	X	
Composites					
Metal-fiber/metal-matrix	X	X	X		
Ceramic		X	X		
Graphite			X		
Ceramics				X	
Control materials				X	
Coatings materials and methods					X

4.0 RESULTS

On the basis of activities performed in FY 1986, an inventory of the materials considered candidates for MMW concept applications and an assessment of the technology status of each material have been developed. A summary of this information will be presented for each of the five generic groups of materials: (1) refractory alloys, (2) composites, (3) monolithic ceramics, (4) reactor control materials, and (5) coatings.

4.1 REFRACTORY ALLOYS

As a result of MMW design efforts performed in FY 1985 and 1986, concept designers have identified a large number of refractory alloys for possible application in MMW systems, see Table 3. In order to aid MMW program participants, a major accomplishment in FY 1986 was the preparation of a refractory alloy bibliography. This bibliography will be issued in mid-November 1986.

Because of the large number of candidate alloys, it is not considered practical to discuss the technical status of each of these alloys. However, the technology status of the following classes of alloys will be reviewed: (1) internally nitrided Mo- and W-base alloys, (2) solution-strengthened Mo- and W-base alloys, (3) solution- and precipitation-strengthened Ta-base alloys, (4) Re and Re-base alloys, and (5) chemically vapor-deposited tungsten alloys. In addition an assessment of the status of these classes of alloys will be provided in the areas of mechanical properties, compatibility with coolants and working fluids, and irradiation effects.

4.1.1 Candidate Refractory Alloys

A summary of the status of technology and of work in progress for the five generic classes of refractory alloys follows.

4.1.1.1 Internally Nitrided Mo and W Alloys. In the late 1960s and early 1970s, it was determined that dilute Mo- and W-base alloys containing hafnium could be internally nitrided, and the resulting HfN dispersion enhanced significantly the high-temperature strength of the alloy. However, little or no data is available on the fabricability, fuel and coolant compatibility, irradiation effects, and long-term thermal stability of these alloys.

Significant progress has been made in FY 1986 to re-establish this technology. During this fiscal year archive specimens of an internally nitrided molybdenum alloy (Mo-1.86Hf) have been utilized to assess the creep properties of these alloys in 1000-h tests. In addition, ingots of Mo-Re have been prepared and will be utilized in FY 1987 to assess the fabricability of the Mo-Hf and Mo-Re-Hf alloys and to optimize the nitriding process. A comprehensive summary of these recent development activities is provided as Appendix A.

Table 3. Summary of Candidate Refractory Alloys by Function Area

Candidate Refractory Alloys	Fuel Cladding	Thermionic Emitters	Reactor Structural: Core Support: Fuel Pins or Support	Reactor Structural: Core Support: Frit (BNL) Exit	Reactor Structural: Core Support: Frit (BNL) Inlet	Reactor Structural: Pressure Vessel	Nonreactor Structural: Piping and Heat Exchangers	Nonreactor Structural: Turbine Housing	Nonreactor Structural: Turbine Rotor and Stators	Nonreactor Structural: Radiators
CVD-W		✓								
Internally nitrided Mo	✓		✓			✓	✓	✓	✓	
Internally nitrided W	✓	✓	✓			✓	✓	✓		
Mo-Re	✓		✓			✓	✓	✓	✓	
Mo-TZM			✓			✓	✓	✓	✓	
Re				✓						
Ta	✓		✓			✓	✓	✓		
Ti										✓
V										✓
W-Re	✓	✓	✓	✓		✓	✓	✓		
Zr					✓					

4.1.1.2 Mo- and W-Base Alloys. The improved low-temperature ductility and increased high-temperature strength of dilute Mo- and W-base alloys containing rhenium was noted in the early 1970s. In FY 1985 insufficient data was available to assess their fabricability, long-term load-carrying capability, tolerance to irradiation effects, and compatibility with candidate fuel and coolants. As part of the SP-100 Project, Los Alamos National Laboratory (LANL) successfully fabricated Mo-Re alloys into sheet and tubing. Creep test specimens from sheet stock made from this alloy were tested at ORNL. Information from the creep tests is summarized in section 4.1.2, "Load-Carrying Properties." No work on Mo-Re alloys funded by the MMW program was performed in FY 1986.

Also in support of the SP-100 Project, ORNL fabricated a W-25Re alloy ingot into sheet. Specimens of both the W-25Re and the Mo-Re alloys were irradiated in the materials irradiation test B-351. Because the reference structural material for the SP-100 Project was later determined to be Nb-1Zr, no postirradiation evaluation (PIE) of the Mo- and W-base alloys was performed. However, PIE of both alloys is a recommended FY-1987 MMW materials program activity. Completion of this evaluation will provide valuable information on Mo- and W-base alloys.

Activities were initiated in FY 1986 as part of the MMW Program to assess W-Mo-Re alloys. The principal focus of these activities to date is the laboratory preparation of a number of candidate W-Mo-Re alloys for subsequent study.

4.1.1.3 Ta-Base Alloys. Extensive Ta alloy development was performed in the 1960s and 1970s and led to the Ta-W-Hf-Re-C alloy (ASTAR 811C). The substantial mechanical properties data base that exists for this alloy indicates the alloy's excellent long-term load-carrying capability at temperatures to 1600 K. Additional data are needed on fabrication of required product forms, irradiation effects, and alloy compatibility with candidate fuels and coolants and optimization of thermomechanical treatment for long-term (7-year) creep strength.

Activities were initiated in FY 1986 as part of the MMW program to fabricate ASTAR 811C tubing. Successful completion of this activity in early FY 1987 will provide needed information regarding the fabricability of this alloy.

4.1.1.4 Re-Base Alloys. The primary application for Re alloys in the MMW Program is the high-temperature frit used in the Brookhaven National Laboratory (BNL) particle-bed-core/Brayton-cycle concept. Relative to this application, little or no alloy development has been performed on Re or Re-base alloys; however, preliminary compatibility data developed by BNL suggest that rhenium alloys can tolerate hydrogen exposure at the temperatures proposed for bimodal Brayton cycle concepts. More recent work by BNL revealed interactions between rhenium and the SiC coating of HTGR triso fuel. Compatibility information for the ZrC coating for the proposed fuel for the BNL concept is needed.

Work is under way by the SP-100 Project to procure commercially small-diameter thin-wall Re tubes produced by the chemical vapor deposition (CVD) process. For the SP-100 Project these tubes will be evaluated for possible use as barriers between the UN fuel and the Nb-1Zr fuel cladding.

Experience in the fabrication of these fuel barriers will provide valuable information relative to the fabricability of the material. Work was initiated in FY 1986 by Babcock and Wilcox to procure rhenium tubing from commercial sources and machine the slots required for the hot frit application for the BNL concept. At this time, no material has been provided for evaluation. Further, no information is available for rhenium regarding the high-temperature load-carrying properties and irradiation effects.

4.1.1.5 Chemical Vapor Deposited Tungsten. In the period from 1963 to 1970 extensive efforts were made to develop the technology base for complex tungsten thermionic emitters fabricated by chemical vapor deposition (CVD) methods. At present the operational temperature limits for CVD tungsten emitters appears to be 1800 to 1900 K for continuous operation because of the limited creep strength of the CVD tungsten at those temperatures. Development of CVD methods to fabricate tungsten alloyed with rhenium or other strengthening additions were originally thought to be a means to allow operation of thermionic emitters in the steady state or burst mode at temperatures significantly higher than is possible with CVD tungsten emitters.

Again, work on the SP-100 Project in the fabrication of fuel cladding barriers has indicated that W-5Re thin-walled tubing commercially produced was not of satisfactory quality for fuel barriers or anticipated thermionic emitter applications. However, alternative CVD methods available at Los Alamos National Laboratory may provide a method for improving CVD W-5Re tubing. No CVD tungsten activities funded by the MMW program were performed in FY 1986.

4.1.2 Load-Carrying Properties

MMW concepts are being designed to operate at temperatures in excess of 1400 K to minimize the mass and volume of the power system. Information on the steady-state load-carrying capabilities of the refractory alloys considered as candidates for MMW structural applications can be estimated by measuring the creep strength and minimum creep rates at temperatures above 1300 K.

For past reactor designs a total creep strain of 1 to 2% was considered to be the limit for fuel cladding or structural applications. Until design criteria are established that provide quantitative strain limits for the components of a MMW system, estimates of the stress required to produce 1% creep strain in seven years are being used to provide a relative ranking of the elevated-temperature strength of the candidate alloys. Such analysis has been performed using the Larson-Miller parameter for those alloys thought to have sufficient creep data to allow extrapolation to seven years of operation. A comprehensive creep properties data base was prepared during FY 1986 and is scheduled to be issued in December 1986.

On the basis of this analysis, only the data bases for T-111 and ASTAR 811C are considered adequate to support the detailed design of a MMW system at this time. This analysis indicates that the creep strength of ASTAR 811C exceeds that of all other candidates at temperatures to 1600 K. Also the creep strength of W-5Re exceeds that for CVD-W and W-25Re for all temperatures of interest for MMW systems. The internally nitrided Mo-base alloys and the W-Mo-Re alloys have not been included in this analysis because the available creep property data is considered insufficient to allow extrapolation.

In addition to creep properties, other mechanical property considerations will also be needed to assess the feasibility of candidate materials. These additional considerations include fatigue, creep-fatigue interactions, fracture toughness, and cyclic crack growth. At present no information is available regarding these properties for the candidate materials.

4.1.3 Compatibility

The long-term (>10,000 h) compatibility of candidate refractory alloys with alkali metals (liquids and vapors), inert gases, and hydrogen at the proposed MMW operating temperatures is not known sufficiently to gauge reliable performance of these materials. Resolution of these issues is critical to both MMW concept and material down-selection decisions.

4.1.3.1 Inert Gas Environment. Among the prospective environments for MMW concepts, He is the most difficult to assess in terms of refractory-metal compatibility. The available test data are totally consistent in showing unacceptable oxidation rates for Nb- and Ta-based alloys in He at the impurity levels achievable by today's technology. Mo- and W-base alloys are much less sensitive to oxidation by small concentrations of impurities in He than are Nb- and Ta-base alloys.

A major feasibility issue associated with the operation of a power conversion system using inert gases and refractory alloys is the degradation of the alloys from the impurities in the gas and the mass transport of alloy impurities from hotter to cooler alloy surfaces. To assess the relative compatibility of candidate refractory metals for inert gas applications, gas-metal reaction studies performed in vacuum and helium environments containing controlled concentrations of oxidizing and carburizing gaseous impurities are required. During FY 1986, efforts were initiated to design and refurbish the equipment necessary to perform the needed tests.

4.1.3.2 Hydrogen Gas Environment. The response of candidate refractory alloys in the hydrogen coolant proposed for use during the burst power phase of some candidate concepts must be assessed. On the basis of past experience, the tantalum alloys are known to be prone to hydrogen embrittlement. Preliminary studies of the compatibility of tungsten and rhenium materials were performed by BNL early in FY 1986 and indicated encouraging response for the rhenium material. Because of the short-term nature of these tests, additional tests are needed to confirm these observations.

4.1.3.3 Alkali Metal Environment. The evaluation of materials for lithium reactor systems has progressed through screening corrosion tests of Nb-, Ta-, Mo-, and W-base alloys to loop testing of Nb- and Ta-base alloys up to 1473 K and finally to forced corrosion and engineering loop systems of the T-111 alloy in the temperature range from 1373 to 1640 K. The mass transfer properties of tungsten and molybdenum alloys in lithium have not been measured under forced flow conditions. However, both alloy systems appear highly corrosion resistant to lithium based on isothermal tests.

Corrosion data relating the use of refractory metals for containment of boiling alkali metals are more extensive for potassium than sodium and are about equally divided between tantalum and niobium alloys. Both alloys have been shown to be highly corrosion resistant to sodium and potassium under boiling and condensing conditions up to 1373 K. Further, long-term tests of Mo-TZM nozzle-blades assemblies have demonstrated excellent resistance of this alloy to erosion-corrosion in saturated potassium vapor. To assure the feasibility of candidate alloys currently being considered for MMW applications, testing is required to evaluate the purity, fabrication, and heat treatment requirements of these candidate alloys. During FY 1986, efforts were initiated to refurbish the equipment necessary to perform the needed tests.

4.1.4 Irradiation Effects

Irradiation effects information on the candidate alloys for space reactor applications at relevant irradiation and test conditions is almost nonexistent. The few existing data on refractory metals are either for unalloyed metal or for simple alloys. The guidance provided by experience with stainless steel and other metals will not be adequate to assess the behavior of the candidate alloys during service.

Many of the alloys being considered for MMW applications were included in SP-100 irradiation experiments but were never evaluated after testing. Although the fluence and operating temperatures for these experiments were lower, in some cases, than those required for MMW applications, postirradiation examination (PIE) of these alloys will greatly expand the irradiation effects information available.

In FY 1986 activities were initiated for performing the PIE of those specimens previously irradiated by the SP-100 Project. In addition, preliminary scoping studies of the irradiation experiments necessary to obtain the needed materials performance information was initiated. These scoping studies thus far have been limited to testing in the Fast Flux Test Facility (FFTF). A summary of these coping studies are provided as Appendix B.

4.2 COMPOSITES

Several innovative and developmental composite systems offer the opportunity for improvements in MMW system performance. These candidate composite systems include carbon/carbon (C/C), ceramic, and metal-fiber/metal-matrix.

4.2.1 Carbon/Carbon Composite

Effective integration and advancement of current C/C composite technology will provide the opportunity for significant improvement in MMW system performance. Concept designers have identified roles for C/C composites in gas turbine applications as flexible membranes for balloon radiators and as flywheels for energy storage applications. With regard to these turbine applications the current technology for C/C composites will draw on the following programs. (1) The DOE automotive flywheel program utilizes glass/epoxy hubs surrounded by an interference-fit graphite-fiber/polymer composite ring. Rim structures 38 cm in diameter utilizing uniaxial hoop-wound fibers and both hard and soft binder matrices have been spun to 45,000 rpm. (2) The DOD has developed C/C composites for turbopumps and generator applications that are approximately 25 cm in diameter. These structures, consisting of 2- and 3-D C/C composite structures attached to a metal hub, have been spun to 50,000 rpm. (3) The DOD is building the technology base for the design and fabrication of large-diameter (>24 cm in diam) C/C composite rotors and stators for cruise missile engines. (4) The DOE centrifuge program utilizes large-diameter cylindrical polymer/fiber composite structural components. These components have been designed and spun at high speeds successfully.

To meet the performance objectives of the MMW program, improvements are required in the areas of fiber surface morphology and chemistry, chemical modification of the precursor matrix materials, and fabrication techniques. In addition significant effort will be required in developing coatings to protect the composite from the Brayton cycle working fluid. (C/C composites are not considered candidates for Rankine cycle applications because of compatibility considerations.) No work in the area of C/C composites was initiated in FY 1986.

4.2.2 Ceramic Composites

Recent work on the development of ceramic-matrix composites for structural applications has demonstrated that these materials can be fabricated with both high fracture toughness and high fracture strength at elevated temperatures (up to 1500 K). This combination of both high-temperature strength and high fracture toughness is a unique and significant accomplishment in the field of ceramic-matrix composites.

At least five promising ceramic-matrix composite systems will be investigated for Brayton and Rankine cycle applications. Of the five candidate composite systems, two systems are already developed (SiC-whisker-reinforced Al_2O_3 and SiC-whisker-reinforced Si_3N_4) and three systems are speculative (carbon-reinforced ZrC, SiC-fiber-reinforced ZrC, and Al_2O_3-reinforced Al_2O_3).

During FY 1986 the feasibility of using any of the five candidate ceramic composites for Brayton and Rankine cycle concepts was assessed. Two approaches were taken in this assessment: an equilibrium thermodynamic analysis and a nonequilibrium assessment based on a literature review. In the equilibrium thermodynamic analysis, gas partial pressures of gas species formed were calculated for each combination of ceramic composite and power cycle over an appropriate range of temperatures. Three power cycles were considered: the closed Brayton cycle using an inert-gas working fluid with a peak temperature of 1800 K, an open Brayton cycle using hydrogen as the working fluid for short duration (30 min) at operating temperatures to 2500 K, and a Rankine cycle using liquid and vapor potassium as the working fluid with a peak temperature of 1550 K.

Combinations of composite material and power cycle resulting in the formation of a gas species with a partial pressure of 10^{-5} Pa or greater were considered incompatible. Combinations with gas species pressures of 10^{-10} or less were considered compatible. The results of the equilibrium assessment based on calculated gas species pressures are summarized in Fig. 1. Based on this assessment, it is recommended that Al_2O_3-reinforced Al_2O_3 and carbon-fiber-reinforced ZrC be considered for the Brayton cycle continuous mode and that Al_2O_3-reinforced Al_2O_3, SiC-fiber-reinforced ZrC, and carbon-fiber-reinforced ZrC be considered for the Rankine cycle. None of the candidate ceramic composite systems are likely to be sufficiently stable under Brayton cycle burst mode conditions.

4.2.3 Metal-Fiber Metal-Matrix Composites

The development of composite components composed of continuous high-strength W-base alloy (W-Hf-C or W-Re-Hf-C) filaments in a refractory-alloy matrix is an innovative step in meeting the high-temperature structural materials requirements of proposed MMW concepts. The basis for this technology advance is the excellent high-temperature strength and recrystallization resistance of W-base alloys and the excellent compatibility of Nb-, Ta-, Mo-base alloys with the heat transfer media being considered for MMW concepts. Specifically, Nb- and Ta-base alloy matrices are expected to demonstrate excellant compatibility with the alkali liquid metal coolants and working fluids being considered for candidate Rankine-cycle concepts and Mo-base alloys are expected to demonstrate good compatibility with the inert gas coolants and working fluids used with Brayton-cycle concepts.

Although it will be necessary to determine the mechanical properties and characterize the effect of irradiation on metal-fiber/metal-matrix composites, two near-term feasibility issues have been identified. These feasibility issues are associated with the demonstration of the capability to fabricate a composite using a Nb-base alloy matrix and with the assessment of the compatibility of the filament with candidate matrix materials.

4.2.3.1 Process Development. A number of critical issues concerning the fabrication of tungsten-base alloy filaments and their consolidation with the matrix material have been identified. These issues are (1) select the optimum alloy composition for the filament wire, W-4Re-0.35HfC versus W-0.35HfC; (2) demonstrate the successful fabrication of small-diameter tungsten-alloy filaments; (3) determine the optimum technique for cladding tungsten-alloy filaments with niobium-alloy matrix material; and (4) develop consolidation techniques for producing refractory-metal-matrix composites suitable for mechanical properties testing and compatibility studies. Significant progress was made in FY 1986 in installing and qualifying the equipment necessary to fabricate the candidate metal-fiber/metal-matrix composites.

4.2.3.2 Compatibility. One of the factors that affect the long-term life and ultimately the mechanical behavior of these high-temperature composites is the compatibility of the tungsten-alloy filament and the niobium-alloy matrix. Although no deleterious intermetallic compounds are expected to form in this system, a potentially serious concern exists regarding the

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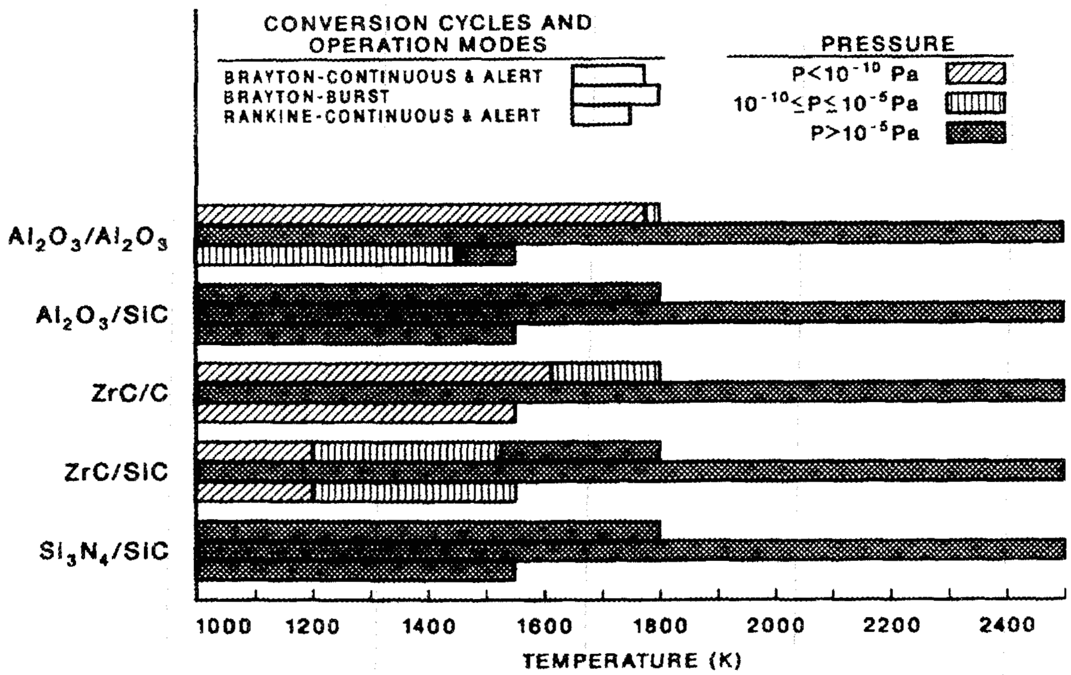

Fig. 1. Thermodynamic equilibrium assessment of ceramic/ceramic composites considered as candidates for the MMW power system applications.

formation of Kirkendall voids. Kirkendall voids occur when two materials having large differences in melting points are intimately joined and held at high temperature for an extended time. This phenomenon could result in interconnected porosity near the filament/matrix interface. It is not known if the Kirkendall void phenomenon will be a factor in the proposed refractory-metal-matrix composites; however, tests to aid in answering this question have been designed, and appropriate specimens are being fabricated.

4.3 CERAMICS

MMW concept developers have identified the use of developmental ceramic materials for in-core reactor structural applications and nonreactor structural applications. Specifically, nonreactor applications include Brayton cycle turbine rotors and stators and Rankine cycle alternator housing applications. Although a description of the candidate materials for these applications follows, no work on monolithic ceramics was performed in FY 1986.

4.3.1 Silicon Nitride and Silicon Carbide

These ceramics are being considered as candidates for structural applications in the hot flow path of advanced Brayton turbine cycles using either helium or hydrogen as the working fluid. A major concern is the critical flaw sensitivity of these materials. The hot flow path components include the rotor, stator, inlet shroud, exhaust housing, and ducting inlet shroud. The current major gas turbine technology program in the United States concerned with the application of Si_3N_4 and SiC is the Advanced Gas Turbine (AGT) Program funded by DOE. Because the turbine rotors required for MMW applications are approximately 4.5 to 8 times larger than the diameter of rotors currently being developed by the AGT program, a significant challenge exists in meeting MMW requirements with Si_3N_4 or SiC. Specific technical concerns include lack of suitable fabrication technology, lack of a long-time mechanical property data base, and lack of a suitable design methodology.

4.3.2 Alumina and Beryllia

Another potential area of application for ceramic materials in MMW systems is the Rankine cycle alternator housing, which is required to isolate the alternator stator from the potassium working fluid. Development activity is needed on these materials to provide the properties required for selection and design of full-scale housings and to demonstrate the feasibility of fabricating prototype ceramic components. In addition, the technology for joining the ceramic alternator housing to the refractory-alloy housing of the turbo-alternator must be developed and demonstrated for the joint sizes consistent with MMW system designs. Preliminary findings obtained in the 1960s included good results for small samples using rapid brazing cycles. Major difficulties were encountered when the joints were scaled to the size required for 100-kW(e) Rankine cycle power systems. These problems were probably due to a lack of understanding and control of the thermal expansion differences between the ceramic, the brazing alloys, and the refractory-alloy component. These problems have not been solved and will be more severe for the much larger turbogenerator components required for megawatt power systems. The demonstration of high-reliability joints between the candidate ceramics (alumina and beryllia) and refractory alloys of a prototype size for MMW systems and compatibility of these materials in a hot potassium environment represents a major unanswered feasibility issue.

4.3.3 Boron Carbide/Beryllium Oxide

A primary in-core structural application for $^{11}B_4C$ and BeO has been identified in the Lawrence Livermore National Laboratory gas-cooled reactor concept. For this application these materials must demonstrate high strength, fracture resistance, and compatibility with candidate fuels and working fluids. For the proposed components, both monolithic and modular fabrication methods must be evaluated.

4.3.4 Thermionic Insulation

Thermionic sheath insulator performance is one of the major life-limiting factors in the in-core thermionic concept, and it can also limit the maximum operating voltage. The environment in which the sheath insulator operates is unprecedented in its severity in terms of the combination of temperature, radiation, and electric fields. Alumina, which has been the standard in earlier programs, may be limited to a lifetime of 3 to 4 years because of radiation effects.

Candidate materials for improved sheath insulators may be classified in two general areas: (1) improved ionically bonded materials such as yttria and yttrium aluminum garnet and (2) covalently bonded materials such as silicon nitride and aluminum nitride.

The ionic materials have the advantage of a thermal expansion coefficient that is compatible with the niobium sheath structure, along with good irradiation performance. The principal concern with their use is their electrical behavior at high electric fields and high temperatures in a radiation environment. The covalent insulators are expected to have excellent insulating properties under thermionic conditions but have a low thermal expansion coefficient that could lead to debonding and poor heat transfer. This thermal expansion mismatch can potentially be mitigated by the use of compliant interlayers or by segmenting the sheath insulator.

4.4 CONTROL MATERIALS

Reactivity control materials (reflectors and absorbers) with the neutronic worth and ability to withstand long-term irradiation and thermal shock requirements for MMW concepts are not currently available. The current state of the art for control materials that have a relevance to the MMW program is embodied in the LMFBR, HTGR, and SNAP reactor programs. Control materials evaluated by those programs hold promise for meeting the MMW program requirements. However, the ability of these materials to tolerate the thermal shock associated with bimodal operation, remain chemically stable in the proposed operating environments, and resist damage during high-flux high-temperature neutron irradiation remains a large uncertainty.

The approach to reactor control for MMW will undoubtedly involve in-core control rods typical of LMR and HTGR designs or reflector control typical of SNAP and SP-100 designs. Combinations of in-core and reflector control components or new innovations are additional possibilities. The control strategy will influence selection of specific materials that have the appropriate combination of nuclear properties, physical properties, chemical stability, and irradiation stability consistent with the application.

LMR and HTGR control materials technology provides a base for assessing in-core applications in terms of MMW requirements. B_4C is the material for which the greatest experience is available in the LMR, and it has been incorporated in boronated graphite in HTGR control rods. Considerations of the ratio of neutronic worth to material weight lead to identification of B_4C as a prime candidate for MMW in-core control systems.

Other materials which have received consideration but only minor development support for LMRs include rare earth compounds such as Eu_2O_3, EuB_6, and metallic Ta. LMR operating conditions involve temperatures hundreds of Kelvins lower than MMW goals, and LMRs use stainless steel rather than refractory metal cladding and sodium rather than lithium coolant. Compatibility of candidate absorber materials with cladding materials and the effect of possible contact with the coolant are obvious feasibility issues. The transients imposed during bimodal operation raise substantial issues regarding the mechanical stability of control materials. For example, B_4C will contain helium from neutron captures at low power operation but would be expected to release the helium at some unknown rate during transient operation. B_4C swelling at steady-state operation is reasonably well characterized, but the effects of rapid increase in temperature and neutron reaction rate are unknown. Much uncertainty exists regarding the mechanical response to thermal stresses in such transient compounds.

Neutron reflector applications, such as rotating drums outside the core in the SNAP and SP-100 concepts, are based on use of beryllium metal or BeO. Total fast neutron exposure and temperatures of operation are significantly higher for MMW concepts than the current experience base. Refractory compounds of beryllium may be required for dimensional stability in the high-temperature MMW systems. Little development work for reactor applications has been done on these materials. Enhanced thermal conductivity and strength of such compounds as Be_2C could be advantageous where reflectors are part of the controls for reactors with burst mode capabilities.

In all cases, candidate materials must be subject to basic screening tests to evaluate the compatibility of these materials with cladding and coolants, thermomechanical stability, and tolerance to neutron irradiation effects. The activities to select and qualify reactivity control materials must be integrated with the irradiation test vehicles developed for the materials and fuels program element.

In FY 1986, no activities in the development of control materials for MMW applications were initiated. However, the design of a FFTF irradiation test of candidate SP-100 control materials is under active consideration by the SP-100 project. Performance of such a test could provide much valuable information to the MMW Program in the near term.

4.5 COATINGS

Development of innovative coatings and coating methods will be required to meet the diverse materials challenges of the proposed MMW reactor concepts. The specific challenges are in the areas of emissivity coatings for radiators, coatings to reduce wear between moving components operating at high temperatures, and methods to improve the electron work function of candidate thermionic emitters. No work on coating development was initiated in FY 1986.

4.5.1 High-Emissivity Coatings

MMW designers may utilize metallic radiators to dissipate waste heat from the energy conversion, instrumentation and control, power processing, and radiation shielding subsystems. Because metals are poor emitters of thermal radiation, they must be coated with a material to increase the emissivity to greater than 0.85. Such a coating must retain high emissivity throughout the life of the radiator and must not degrade the properties of the metallic substrate through chemical interactions.

To meet these requirements, a coating material must have a high intrinsic emitting power, low vapor pressure, thermal expansion characteristics similar to those of the base metal, and low solubility in the substrate material. In earlier work on the SNAP program, oxides such as Fe_2TiO_5, $CaTiO_3$, Cr_2O_3, and platinum-coated Al_2O_3 were applied to Nb-1Zr components. Results of these tests showed serious problems regarding the long-term stability of these materials at high temperatures. The problems included significant degradation of the thermal emittance performance and reactions with the substrate. The available technology for emissivity coatings is not considered adequate for the proposed MMW applications.

A variety of coating techniques are available, including plasma spray, physical vapor deposition, chemical vapor deposition and its variants, and electrochemical systems. Candidate materials will be chosen for their durability and compatibility because improved emissivity will be provided by deposit morphology rather than by inherent material properties. Candidate materials include nitrides, borides, and carbides of titanium or silicon. Intermediate coatings may be necessary to reduce stress due to differences in thermal expansion mismatch between substrate and coating or to protect refractory alloy substrates from chemical interaction with coating gases or coating elements.

Potentially fruitful new approaches have been identified for developing high-emissivity surfaces. One approach involves ceramic coatings with tailored surface morphologies, such as high-relief, faceted surfaces or high-density whisker-containing surfaces. A second approach utilizes a two-phase coating deposited on a radiator surface followed by dissolution of one of the phases in the near-surface region to generate surface porosity that would provide improved emissivity.

4.5.2 Wear of High-Temperature Moving Components

Current MMW concept designs include many moving components operating at temperatures that may range from 600 to 1800 K. These applications include pumps, valves, rotating turbines and alternators, and rotatable neutron reflector and absorber materials for reactor control. Excessive wear of the bearing surfaces associated with these components may significantly limit MMW system performance.

Wear-resistant coatings have been developed for low-temperature commercial applications such as tool bits, bearings, valves, and seals. Materials developed for these applications include TiC, TiN, TiCN, Al_2O_3, TiB_2, Cr_2O_3, partially stabilized ZrO_2, and Si_3N_4 deposited by physical vapor deposition, chemical vapor deposition, or plasma spraying. In addition, higher-temperature coatings currently under development for wear resistance, thermal insulation, and lubrication of advanced heat engine components appear to be attractive candidates for MMW applications.

4.5.3 Improved Electron Work Function

Thermionic energy converters hold the potential for providing effective static and closed-cycle power for MMW applications. A key to higher performance for thermionic conversion systems is development of electrode surfaces with an improved electron work function and a low thermal emissivity. Presently, chemically vapor deposited coatings of tungsten and rhenium are used to obtain work functions near 5.0 eV. Oxygen is used as an additive to give higher effective work functions. In practically all cases, the coatings are applied to refractory metal substrates, typically tungsten.

5.0 CONCLUSIONS AND RECOMMENDATIONS

On the basis of this assessment, no engineering materials currently considered qualified for high-temperature nuclear applications were found to be developed sufficiently to assure meeting the objectives of the MMW program. However, a number of innovative and evolutionary candidate materials have been identified by concept designers and material specialists, and they may have the potential of meeting MMW requirements. Many of these materials offer the potential of reducing MMW system mass significantly and increasing system lifetime.

In general, these recommended materials can be organized into five generic groups: (1) refractory alloys, (2) composites, (3) monolithic ceramics, (4) control materials, and (5) coatings. Although some of the candidate materials are more highly developed than others, significant development efforts will be required to qualify any of these materials for MMW applications.

In order to ensure that the MMW Program objectives will be met, it is recommended that a comprehensive materials program continue. This activity should pursue the development of both innovative and evolutionary materials to ensure that the material(s) needed to enable a MMW concept and allow advancement to the ground demonstration phase of the program are available. This activity must be oriented toward identifying and resolving key feasibility issues associated with specific combinations of candidate materials and concepts through in-reactor tests, compatibility studies, and mechanical and physical properties measurements. The findings of these studies will be used to aid in periodic materials and concepts down-selection decisions leading to the identification of the one concept to advance to the ground demonstration phase of the program.

Specifically, the evaluation of Mo-, W-, and Ta-base alloys should be continued in FY 1987. These evaluations should lead to the identification of a reference alloy for the MMW concept structural applications by FY 1991. Further, work should continue in the evaluation of composite materials. Specifically, the ceramic composites appear to be an enabling technology for concepts utilizing either Rankine or Brayton cycle concepts. Based on funding availability, development of metal-fiber/metal-matrix and carbon/carbon composites should be pursued because those innovative materials systems offer the promise of significant improvement in MMW system performance.

Assessment efforts in the areas of control materials, coatings, and monolithic ceramics should be initiated in FY 1987. The purpose of these efforts will be to review the material recommendations of concept developers in the respective areas and to prepare and implement the development program necessary to assess the feasibility of these materials.

Appendix A

INTERNALLY NITRIDED REFRACTORY ALLOY (INRA) DEVELOPMENT

UCID- 20880

INTERNALLY NITRIDED REFRACTORY ALLOY (INRA) DEVELOPMENT

FY 1986 REPORT

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October 6, 1986

Lawrence Livermore National Laboratory

This is an informal report intended primarily for internal or limited external distribution. The opinions and conclusions stated are those of the author and may or may not be those of the Laboratory.
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FOREWORD

This work was performed as part of the Lawrence Livermore National Laboratory effort in support of the Multi-megawatt Space Reactor Program of the Office of Space Reactor Projects, Department of Energy. This report follows the format requested by the Technical Integration Support Office.

Carl E. Walter
MMW Project Leader
Nuclear Systems Safety Program

INTERNALLY NITRIDED REFRACTORY ALLOY (INRA) DEVELOPMENT

1.0 Summary

Investigation of the creep behavior of internally nitrided refractory alloys (INRA) during FY 1986 was limited by the availability of Mo-1.86 Hf material. (This material had been procured by LLNL ca. 1970.) New material has been procured and was received at the end of September. A preliminary investigation of the welding characteristics of Mo-1.86 Hf alloy (prior to nitriding) was conducted. No cracks were observed in welds made by TIG or E-beam.

Based on earlier findings (Ref. 1), a creep specimen, R1, was internally nitrided under conditions which lowers the hardness jump previously experienced near the surface. This specimen exhibited thermodynamic stability at 1573 K and 48.2 MPa for 1500 h at which time it was removed for examination. The specimen had elongated about 7% at the end of the test. Another specimen, R2, is currently being tested at a higher temperature, 1650 K, and lower stress, 13.8 MPa. No elongation has been detected after 630 h.

Indications to date are that internally nitrided Mo-HfN alloy has high-creep-strength and is stable and fabricable. Because of its relatively low density and chemical compatability with numerous coolants (e.g., lithium, hydrogen), Mo-HfN alloy has considerable potential as a structural material in high temperature space power systems. Since molybdenum is not subject to hydrogen embrittlement, this type of irradiation damage will not be significant when Mo-HfN is used near a nuclear reactor.

Successful development of the Mo-HfN alloy will contribute to the feasiblity and reliablity of generic nuclear space power systems in the near term. Subsequent development of W-HfN in an analogous manner could provide similar advantages in the future.

2.0 Introduction

This task is being conducted within the Materials Technology Area of the MMW Space Reactor Program under the program and budget guidance provided in Ref. 2. That program guidance is paraphrased and annotated below with some modifications to reflect a more precise intent:

a. Continue development of high-creep-strength internally nitrided refractory alloys for high temperature nuclear space power applications. — Molybdenum based alloys are to be developed for use as fast-reactor cladding, piping, pressure vessels, turbine blades, and other high temperature components. (Future work on tungsten based alloys is also envisioned.)

b. Procure ingots of base alloys. — Ingots of Mo-1.86 Hf and Mo-15 Re-1.86 Hf should be purchased, rolled into sheet, and made into test specimens.

c. Optimize internal nitriding process parameters. — Parameters to be considered include cold work, heat treatment, nitrogen pressure, temperature, and time.

d. Conduct tests and measurements. — Metallographic diagnostics, including micrographs and TEM, should be used to characterize alloy structure. Tests should be performed to determine properties such as hardness, tensile strength, and creep strength.

e. Refurbish two creep machines. — Refurbish existing machines to achieve reliable operation and data. (This effort is carried out as a separate task, Creep Behavior Laboratory, also under the Materials Technology Area.)

3.0 Task Description

The objective of the INRA Development task during FY 1986 was to address the issues as stated in the program guidance modified as above. Subtasks were carried out in the manner described below.

A cursory examination of welding was initiated to address the general issue of fabricability into useful components. Welding by TIG, E-beam, and laser methods was performed at LLNL facilities.

Bids for casting one ingot (6.8 kg) each of Mo-1.86 Hf and Mo-15 Re-1.86 Hf and rolling 2/3 of each ingot into sheet (1.3 mm) were obtained from three sources. The lowest bid was selected, and a contract awarded on a best efforts basis.

A few specimens of Mo-1.86 Hf alloy were nitrided at LLNL at conditions inferred to be appropriate based on analysis of previous specimens which had been creep tested.

At the end of FY 1985, specimen A3 was undergoing creep testing as reported in Ref. 1. The load on the specimen, which had no detectable creep, was increased to provide a higher stress and the test was continued to rupture. Subsequently two additional specimens, R1 and R2, have been subjected to creep. Specimen R1 has been removed from the creep machine and specimen R2 is currently undergoing test. (Two reliable creep machines with a computerized data system will soon be available for this work.)

Our only execution of work on this task outside the Laboratory has been the procurement of alloys. All metallographic and mechanical property tests as well as welding were performed at LLNL. We have been in communication with personnel at ORNL on this task. There has not been the need for integration of our work with other laboratories.

4.0 Results

Nitriding
Mo-1.86 Hf creep specimens (e. g. R1 and R2) were internally nitrided at 1683 K in one atmosphere of nitrogen for 90 h after recrystallization at 1773 K for 1 h. Hardness measurements were made at various depths below the surface.

These measurements are compared with previous values for the same material (but not recrystallized) in Fig. 1. The reduced hardness level and hardness peak is apparent.

Creep testing
Creep testing of specimen A3 at 1523 K which began in FY 1985 was continued. After an initial period of about 130 h at 75.8 MPa with essentally no creep as reported in Ref. 1, the stress was increased to 82.7 Mpa. The specimen fractured after a cumulative time of 1140 h. The specimen broke after elongating about 18%. This history is shown in Fig. 2.

A recrystallized specimen, R1, was creep tested for 1500 h at 48.3 MPa. The total elongation was about 7%. Its creep history is shown also in Fig. 2. The purpose of this test was to demonstrate the thermodynamic stability of the HfN precipitate structure. This is discussed further below.

A summary of creep tests conducted to date is given in Table 1. This table is provided as a record of events only. It is not intended to present data which is representative of optimized processing parameters for INRA. The data are strongly dependent on specimen history prior to testing.

Table 1. Creep tests conducted to date. (See text.)

Specimen Number	Temperature K	Stress MPa	Time h	Elong. %	Remarks
A1	1591	69.0	120	7	
A2	1573	69.0	240	7	
A3	1523	82.7	1140	18*	* ruptured
R1	1573	48.3	1500	7	
R2	1650	13.8	630*	0	* continuing

Internal Structure Study
As described in Ref. 1, the only significant structural instability observed in the internally nitrided Mo-HfN alloy during long-term high-temperature creep is the migration of grain boundaries in regions of higher hardness containing smaller HfN precipitates.

Examples of grain boundary migration are shown in Figs. 3, 4, and 5 which are optical micrographs of the structure of specimen R-1 after creep for 1500 h at 1573 K and 48.2 MPA stress. Figure 3 shows a region about 0.1 mm below the specimen surface in the center of the specimen gage length (see Fig. 6) of the specimen where grain boundary migration has produced significant precipitate dissolution and coarsening.

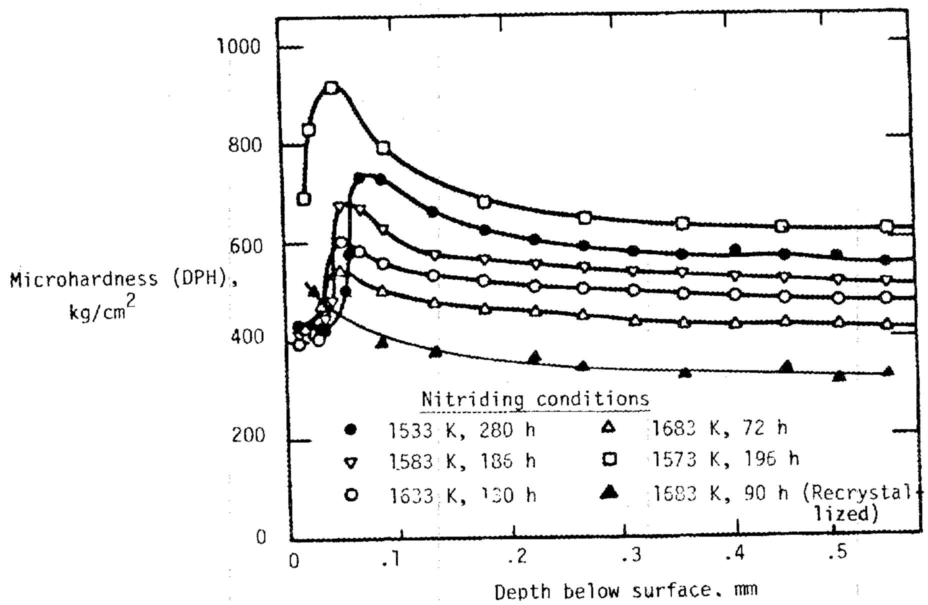

Fig. 1. Hardness vs. depth below surface for several nitriding conditions.

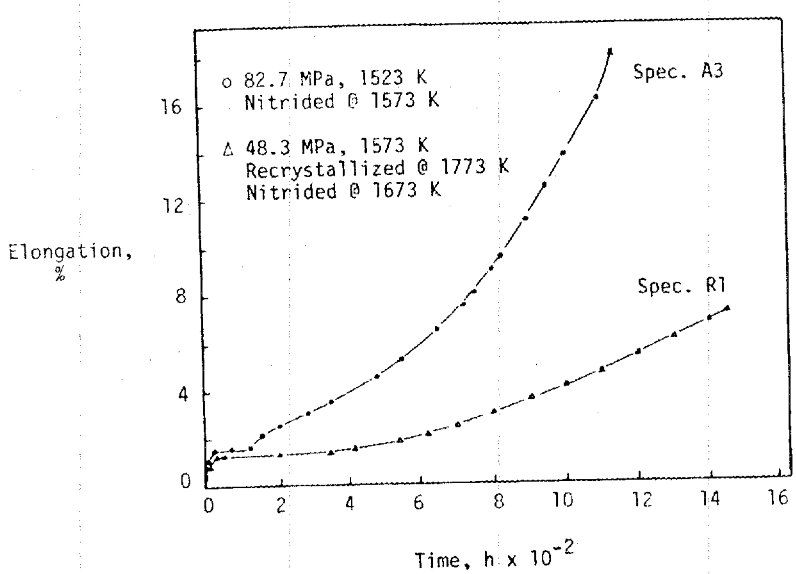

Fig. 2. Creep history for specimens A3 and R1.

Figure 4 shows the structure at 0.1 mm below the surface in a region near the grip section (see Fig. 6) of the specimen, where it can be seen that the extent of grain boundary migration is significantly less than in Fig. 3. The measured temperature difference in the specimen at these two positions during the creep test was about 10 K, whereas the stress differed by about a factor of two. Thus, it appears that the extent of grain boundary migration and associated structural changes at high temperatures are dependent on the applied stress.

Figure 5 shows the structure after creep at a depth of about 0.6 mm below the specimen surface where it can be seen that the HfN precipitate size is larger than that seen in Figs. 3 and 4 and evidence of grain boundary migration is only occasionally observed and is associated with smaller grains.

Welding Study

Preliminary studies of the welding behavior of unconstrained 1.3 mm thick sheet of unitrided Mo-1.86 HF alloy (not nitrided) were carried out by making butt welds and weld beads using TIG, E-beam and laser techniques. Figures 7, 8 and 9 show examples of the cross section microstructures produced in TIG, E-beam and laser welds respectively. In general, sound welds free of cracks and internal voids could be consistently produced by TIG and E-beam techniques. Laser welds were often cracked and contained voids in the fusion zone. The size of the fusion and heat affected zones and the resultant grain size in these zones was substantially larger in the TIG welds than the E-beam and laser welds. No evidence of second phase precipitation in the fusion or heat affected zones was found.

Fig. 3. Cross-section microstructure at 0.1 mm below surface in gage section of specimen R-1. (1000X)

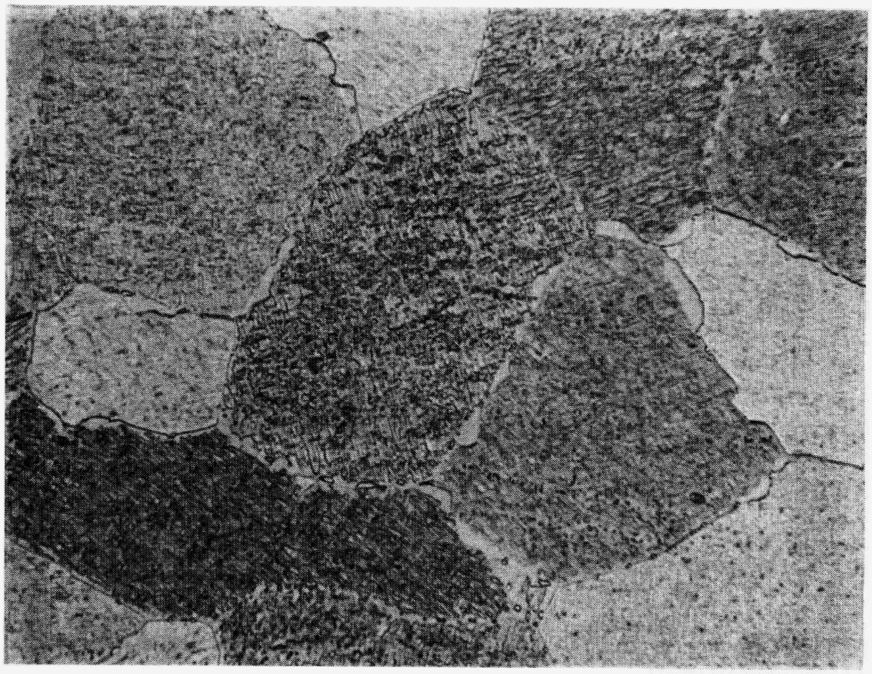

Fig. 4. Cross-section microstructure at 0.1 mm below surface in grip section of specimen R-1. (1000 X)

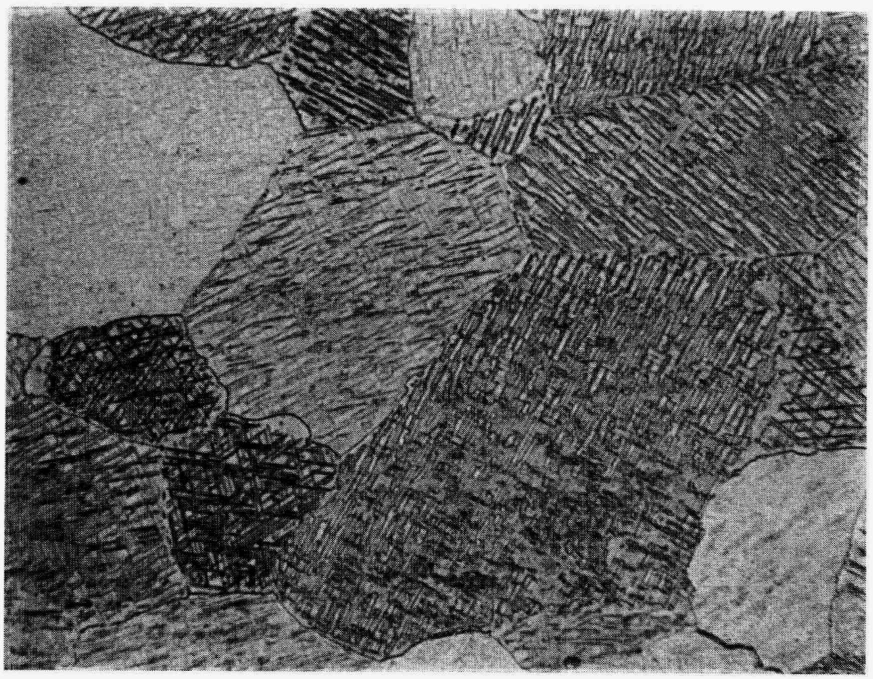

Fig. 5. Cross-section microstructure at 0.6 mm below surface in gage section of specimen R-1 (1000X)

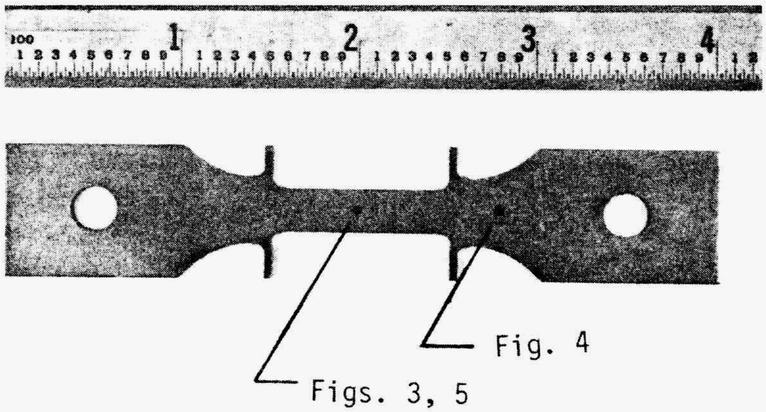

Fig. 6. Tensile creep specimen showing positions corresponding to Figs. 3, 4, and 5.

Fig. 7. Cross-section microstructure of TIG weld in un-nitrided Mo-1.86 Hf.

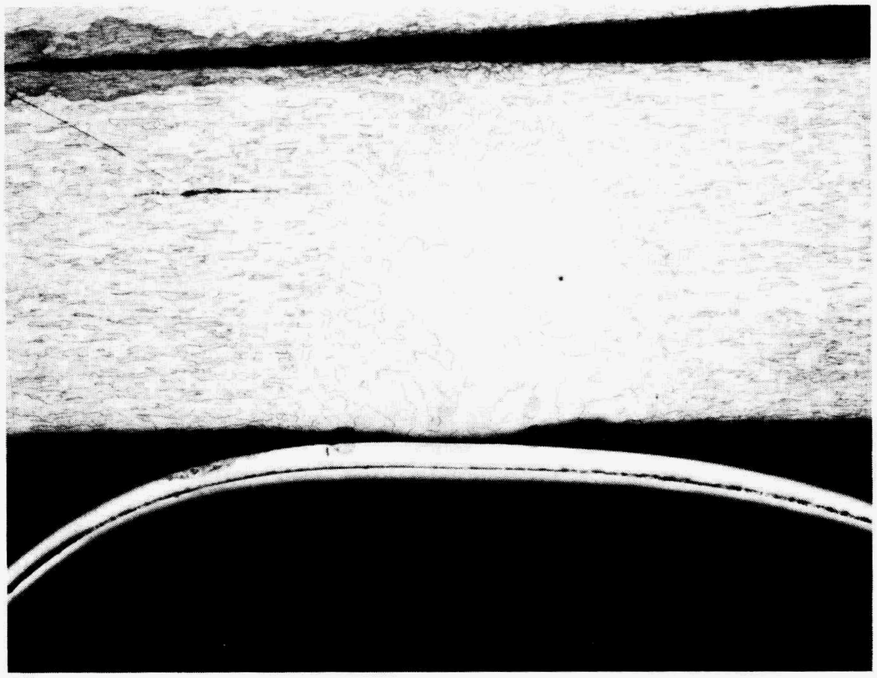

Fig. 8 Cross-section microstructure of E-beam weld in un-nitrided Mo-186 Hf.

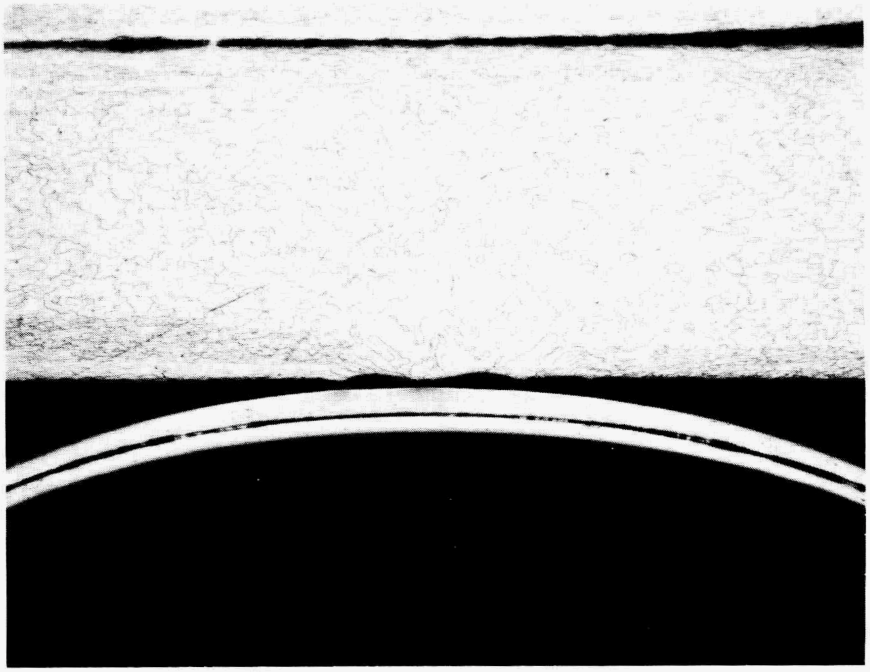

Fig 9. Cross-section microstructure of laser weld in un-nitrided Mo-1.86 Hf.

Alloy Processing

AMAX Materials Research Center was awarded a contract to supply 1.3 mm thick sheet of Mo-15 Re-1.86 Hf alloys each weighing 4.5 kg. The alloys were vacuum arc-cast, extruded, and rolled. AMAX had no previous experience with these compositions. The alloy containing rhenium is of interest in enhancing room temperature fabricability, should that become necessary. The processing procedures and results are described below.

A Mo-1.86 Hf alloy electrode weighing 11.5 kg was prepared by blending molybdenum and hafnium hydride with 400 ppm of carbon to act as a deoxidant. A Mo-15 Re-1.86 Hf alloy electrode weighing 13 kg was prepared by blending molybdenum, hafnium hydride and rhenium powders with 650 ppm of carbon, again as a deoxidant. The blended powders were cold isostatically compacted in rubber molds to form bar electrodes approximately 55 mm diameter. The electrodes were sintered under a partial argon atmosphere and furnace cooled. Carbon and oxygen contents of the sintered electrodes were determined from samples taken from near the half-radius position of the top and bottom of each electrode, and the results are presented in Table 2. Both the carbon and oxygen contents are high, indicating that hafnium carbide and hafnium oxide formed during sintering.

Melting was performed according to standard arc-melting procedures. The arc melting was conducted at a chamber pressure of less than 10 Pa. Ingots approximately 150 mm long and weighing about 10.5 kg were obtained.

The ingots were machined to 80 mm diameter in preparation for extrusion and to remove surface roughness, and were then inspected radiographically to locate internal defects. The shrinkage cavity portions at the upper end of each ingot were removed and defect-free extrusion billets approximately 125 mm long were obtained. Each ingot was then extruded to a 25 x 50 mm rectangular bar.

The carbon, oxygen, nitrogen, hafnium and rhenium contents of the extruded bars were determined, and the results are presented in Table 3. The high carbon and oxygen contents suggest that hafnium carbide and hafnium oxide are present in the arc-cast and extruded bars. Clearly, less carbon should be used in future preparation of alloys of these compositions.

Lengths suitable for rolling were cut from the extruded bars, and surface defects were removed by machining. The rolling blanks were hot cross rolled (rolling direction is perpendicular to the extrusion axis) to 19 mm thickness. The rolling blanks were further hot cross rolled to 1.8 mm thickness. Surface oxides were removed in a caustic bath, and the plates were then cold rolled at room temperature to 1.3 mm thickness (30% cold reduction) and stress relieved. Two sheets measuring approximately 180 by 750 mm were produced for each alloy. The rolled sheets were received at LLNL on September 23, 1986.

Table 2. Carbon and Oxygen Contents of Sintered Electrodes. (Electrodes 78 and 79 were used to produce ingots H-436 and H-437 respectively.)

Electrode No.	Location	Carbon ppm	Oxygen ppm
78	Top	647	418
78	Bottom	453	248
79	Top	823	1081
79	Bottom	810	729

Table 3. Chemical Compositions of Arc-Cast and Extruded Bars

Ingot No.	Carbon ppm	Oxygen ppm	Nitrogen ppm	Hafnium wt%	Rhenium wt%
Goal	50	30	10	1.86	15.0
H-436	264	80	29	1.81	—
H-437	217	82	14	1.82	14.9

5.0 Conclusions and Recommendations

Conclusions

a. Indications to date continue to support the potential of INRA.

b. Internal structure studies show that by controlling grain size and amount of cold work, the results of the nitriding process can be modified. A uniform hardness can be obtained by properly controlling the nitriding parameters. The ability to control nitrogen pressure during the process over a broad range, including above one atmosphere is expected to provide greater uniformity of hardness.

c. Limited welding efforts have produced sound welds using TIG and E-beam techniques in Mo-1.86 Hf alloy sheet. Fabrication of space power components thus appears to be achievable.

d. Alloy compositions Mo-1.86 Hf and Mo-15 Re-1.86 Hf have been successfully produced in sheet form. Additional effort is required to reduce carbon, oxygen and nitrogen impurities.

e. Creep resistance of Mo-HfN alloy is 100 to 1000 times greater than that observed for other molybdenum based alloys. Greater design flexibility yielding lighter and more reliable components would be available with this material.

Recommendations
We propose to address three facets of Mo-HfN alloy development during FY 1987 within funding constraints:

a. Continue evaluation of nitriding parameters and resulting structural properties of the Mo-1.86 Hf alloy. A comparison will be made of the response of Mo -15 Re1.86 Hf with the base alloy to ascertain if their behavior is similar.

b. Fabricate small diameter tubing typical of fuel cladding. This will allow continued assesment of fabrication characteristics and also provide tubular specimens for biaxial (pressurized) creep tests which can be conducted at a much higher density in a high temperature furnace.

c. Evaluate welding parameters and the resulting properties of sheet and tubing.

6.0 References

1. J. B. Mitchell, Creep Behavior of Internally Nitrided Mo-Hf Alloy, Lawrence Livermore National Laboratory Report UCID-20583, October 1985.

2 Letter L. C. Wilcox (DOE-NE) to R. A. DuVal (DOE-SAN) FY 1986 Programmatic and Funding Guidance — Multimegawatt Power Systems, March 28, 1986.

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Appendix B

FY 1986 STATUS REPORT ON MULTIMEGAWATT MATERIALs IRRADIATION ACTIVITIES

Prepared November 1986

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Under Contract No. DE-AC06-76FF02170

SUMMARY

The use of the FFTF reactor for conducting materials irradiations in support of the MMW project has been preliminarily explored. The reactor offers the opportunity of meeting many of the anticipated test environment requirements for the MMW program. The MOTA irradiation vehicle will be capable of providing temperature controlled irradiations to 1250°C and neutron exposures up to 7.3 x 10^{22} n/cm^2. A modest design effort would be required to increase the operating temperature to 1350°C. For higher irradiation temperatures, significant development will be required. The schedule for initiating MMW materials testing in FFTF requires the decision to proceed and funding one year prior to the start of irradiation. If the necessary test environment requires a significant design effort, then up to two years should be allowed for the design and fabrication effort.

INTRODUCTION

The Multimegawatt program is currently considering over a dozen different reactor concepts to supply power in the range of several to hundreds of megawatts. These designs include several fuel types with coolant environments that range from boiling potassium at 1450K (1177°C) to flowing hydrogen gas at 2100K (1827°C). Liquid lithium and mixtures of various inert gasses are also being considered as coolants under certain circumstances. The MMW reactors certainly will be called upon to deliver large amounts of power for short periods but they may also need to be designed to undergo years of low power and months of intermediate power operation prior to the need for burst power. Reactor testing of high temperature cladding/structural materials is required because of the lack of irradiation data on existing and developmental materials. Controlled environment irradiation testing of shielding and control materials is also necessary.

An irradiation test facility should provide a test environment that approximates the anticipated environment for a given Multimegawatt (MMW) reactor concept. This test environment should match materials and coolants to explore chemical compatibility issues, have a similar neutron flux, and cover the appropriate range of irradiation temperatures. Moreover, the irradiation temperature should be controlled to support the engineering data base needed for the reactor designs. At present, many of the proposed reactor concepts raise material performance issues which will require materials testing and in some instances material development. Presently, only limited information exists on the neutron irradiation effects on high temperature refractory alloys. Therefore, to provide the needed data, the irradiation test facilities must provide a sufficiently large test volume.

For reactor concepts requiring testing in a fast neutron environment, the Fast Flux Test Facility (FFTF) at Hanford offers the opportunity of meeting the required test conditions. The Materials Open Test Assembly (MOTA) is a versatile, instrumented irradiation vehicle designed and operated by the U.S. Liquid Metal Reactor materials research program. The MOTA can be modified to meet in-pile testing requirements of the MMW materials program. This report outlines the existing and near-term capabilities associated with the MOTA and identifies some areas which will require further development in fast neutron irradiation testing. Also included is the current reactor schedule and the anticipated timing required to initiate an irradiation test in the FFTF. This information can then be utilized by the MMW project to identify when decisions must be made regarding irradiation testing in the FFTF.

FAST REACTOR TESTING CAPABILITIES

PRESENT MOTA

The MOTA is a versatile, instrumented irradiation vehicle presently utilized by the Liquid Metal Reactor, Fusion, and Thermionic Fuel Emitter (TFE) materials programs. A schematic of the in-core section of the MOTA is shown in Figure 1. The MOTA test train consists of five levels of canisters axially distributed over the core height. Each level has six canisters supported by the central structural tube. The present MOTA has the capability of having a below core canister assembly and up to three levels of canisters (eighteen canisters) located above the core (not shown in Figure 1). Present instrumentation capabilities include forty-eight thermocouple leads and thirty gas lines. The thermocouples are used to monitor and provide feedback to control the temperatures within selected canisters. There are two types of canisters in the present MOTA: weeper and gas-gapped. Both are fabricated from 300 series stainless steel. The weeper type canister allows reactor sodium to flow directly through the test specimen region; the temperature is simply monitored and not controlled. The gas-gapped type canister has an annular gas gap around the specimen region. Reactor sodium flows very slowly through the canister. The specimen temperature is controlled by monitoring the canister temperature and changing the helium-argon gas mixture in the gas gap when the temperature deviates from its control band (±5°C). These gas-gapped canisters have a usable, annual specimen volume of 1.06 in (OD) by 0.18 in (ID) and approximately 5.0 in length. This corresponds to a specimen volume of approximately 4.3 cubic inches.

The present MOTA offers the following testing environment. The specimens are exposed to reactor sodium within the canisters. (Specimens are encapsulated when direct exposure to sodium is a problem). The MOTA is normally loaded into an FFTF row 4 core position which has a peak total neutron flux of 4.7×10^{15} n/cm^2/s. The peak fast neutron flux is 2.8×10^{15} n/cm^2/s (E > 0.1 MeV). The achievable irradiation temperatures and anticipated fast neutron fluence accumulations for the MOTA are given in Figure 2. This information is based upon the FFTF operating at ~300 MWth and a MOTA irradiation cycle lasting 300 effective full power days (efpd). Nominal irradiation temperatures range from 370 to 800°C. (The TFE program is currently operating its canisters at 800°C.) The peak accumulated fast neutron fluence in the MOTA is anticipated to be approximately 7.2×10^{22} n/cm^2 (E > 0.1 MeV). The irradiation period for the MOTA is typically one year.

NEAR TERM MOTA CAPABILITY

The space power SP-100 project is developing the capability of high temperature irradiations (>800°C) within the MOTA irradiation vehicle. A schematic of this canister design is shown in Figure 3. The outer canister is the standard MOTA design and is made from 316 SS. The inner subcapsule is fabricated from Nb-1Zr. The inner subcapsule and the specimen region are isolated from reactor sodium. For SP-100 irradiations the specimen region will be filled with isotopically enriched lithium (0.999 lithium-7). Spinel ($MgAl_2O_4$) spacers will be used to center the inner subcapsule with respect to the outer canister. High temperature, irradiation resistant thermocouples are being developed to provide temperature monitoring and control. A candidate thermocouple design would utilize a Pt-Mo thermocouple wire with BeO insulators and either a Re or Mo-48Re sheath. Based upon neutronic calculations Pt-Mo thermo-element wire should not undergo significant transmutation and therefore should be fairly stable in the neutron flux.

The SP-100 canister design will provide controlled irradiation temperatures of 1000 to 1250°C. The upper temperature limit is dictated by the strength of the Nb-1Zr inner subcapsule. Though the thermocouple may have an operational range to 1500°C, large stress-induced strains due to helium gas accumulation within the subcapsule limits the upper operating temperature for this material. This temperature limit could be increased if a different material such as TZM (a molybdenum based alloy) were used. A modest development effort would be required to increase the operating temperature to approximately 1350°C. These Nb-1Zr and TZM subcapsules may have a lower bound operating temperature due to swelling. The peak swelling temperature in Nb and Mo is in the range of 800-1000°C. Swelling data on these candidate materials would be helpful in assessing this potential limit.

ANTICIPATED TESTING DEVELOPMENT

Present and near term testing capabilities in the MOTA are thus far limited to irradiation temperatures to 1350°C, fast fluences to 7.2 x 10^{22}

n/cm^2 ($E > 0.1$ MeV), and a lithium environment. Present MMW conceptual reactor designs have material operating temperatures up to 1800°C, other working fluids such as potassium, helium and hydrogen, and potential high neutron fluence exposures. The extension of irradiation testing capabilities to these environments will require significant development.

Irradiation temperatures above 1350°C will require the use of high temperature subcapsule materials other than Nb- or Mo-based alloys. At present the W-based alloys look attractive, but development work in the area of product form availability and machining/welding will be required. These elevated temperatures will also require an assessment of reactor safety issues which would include material compatibility and accident scenarios where the reactor coolant is directly exposed to these elevated temperatures.

Fluence exposures greater than 7.2×10^{22} n/cm^2 ($E > 0.1$ MeV) will require specimen reconstitution capabilities similar to those presently used with MOTA in order to efficiently utilize the anticipated MOTA schedule (see the next section of this report). The present design for the SP-100 canisters is not amenable to a rapid reconstitution sequence. A reconstitutable design can be provided, but will require development. Also, new disassembly/reassembly equipment for the hot cells will have to be developed and built.

The SP-100 project will provide radiation resistant thermocouples which should be capable of operating to 1500°C. To obtain reliable and accurate temperature monitoring to very high temperatures, development work will be required. A potential candidate for extended temperature capability is the Nb-Mo thermo-element wire system which should be radiation resistant based upon neutronics calculations. However, Nb-1Mo thermocouple grade wire is not currently available commercially. While the SP-100 experiment in the MOTA will provide information on the radiation resistance of the Pt-Mo thermocouple for temperatures to 1227°C, their performance to higher temperatures will not be obtained in an irradiation environment. Moreover, the irradiation performance of any developmental thermocouple will not be known for a fast neutron environment. Therefore, testing of thermocouples in an irradiation environment prior to any MMW materials is strongly recommended to ensure their performance during actual MMW materials irradiations. The conceptual design for such a thermocouple performance test has been developed for testing in the MOTA irradiation vehicle. This test would provide some statistical and performance data on the effects of fast neutron irradiation and temperature on candidate thermocouple designs.

IRRADIATION TEST SCHEDULING CONSTRAINTS

The MOTA is a multi-purpose irradiation vehicle designed to take advantage of the FFTF reactor schedule. The latest version of this schedule is shown in Figure 4. While these schedules are subject to change, they provide insight into the FFTF operations. The present FFTF schedule calls for ~300 efpd of operation at full power before a significant shutdown period occurs for maintenance and refueling. During this period the MOTA is typically removed from the reactor, the specimens removed and reconstituted into new hardware, and the new assemblies inserted into a new MOTA irradiation vehicle. The irradiation period is typically one year. It also takes approximately one year to fabricate a new MOTA irradiation vehicle. For example, if one wished to begin irradiation in FFTF cycle 11 tentatively scheduled to begin irradiation in April, 1989, then fabrication of that irradiation vehicle would have to begin in April, 1988. Before vehicle fabrication can begin, the specimen test matrix would have to be defined and the irradiation test environment identified. Since the MMW specimens will probably be sealed into the canisters prior to shipment of the MOTA to the FFTF, the specimen receipt at HEDL will probably be required six month prior to the shipping date (i.e., August, 1988). This schedule is based on the use of existing MOTA design and operational capabilities. Test environment requirements which dictate design modification to the MOTA will require an additional lead time to complete the design. At present it is difficult to accurately assess the length time needed for this design effort without knowing the specific test environment requirements. Based upon the effort required to design the SP-100 canisters, a design effort of similar magnitude would take approximately one year. Therefore, if MMW materials irradiation testing were requested to begin with FFTF cycle 11, then decisions to begin such testing must be made this fiscal year.

FIGURE CAPTIONS

(1) Schematic of the present MOTA test train.

(2) Anticipated Fluence Accumulations and Operational Temperatures for MOTA during FFTF cycles 9-11.

(3) Schematic of the SP-100 MOTA canister design which is capable of irradiation temperatures to 1250°C with a static lithium environment.

(4) FFTF reactor schedule as of October, 1986.

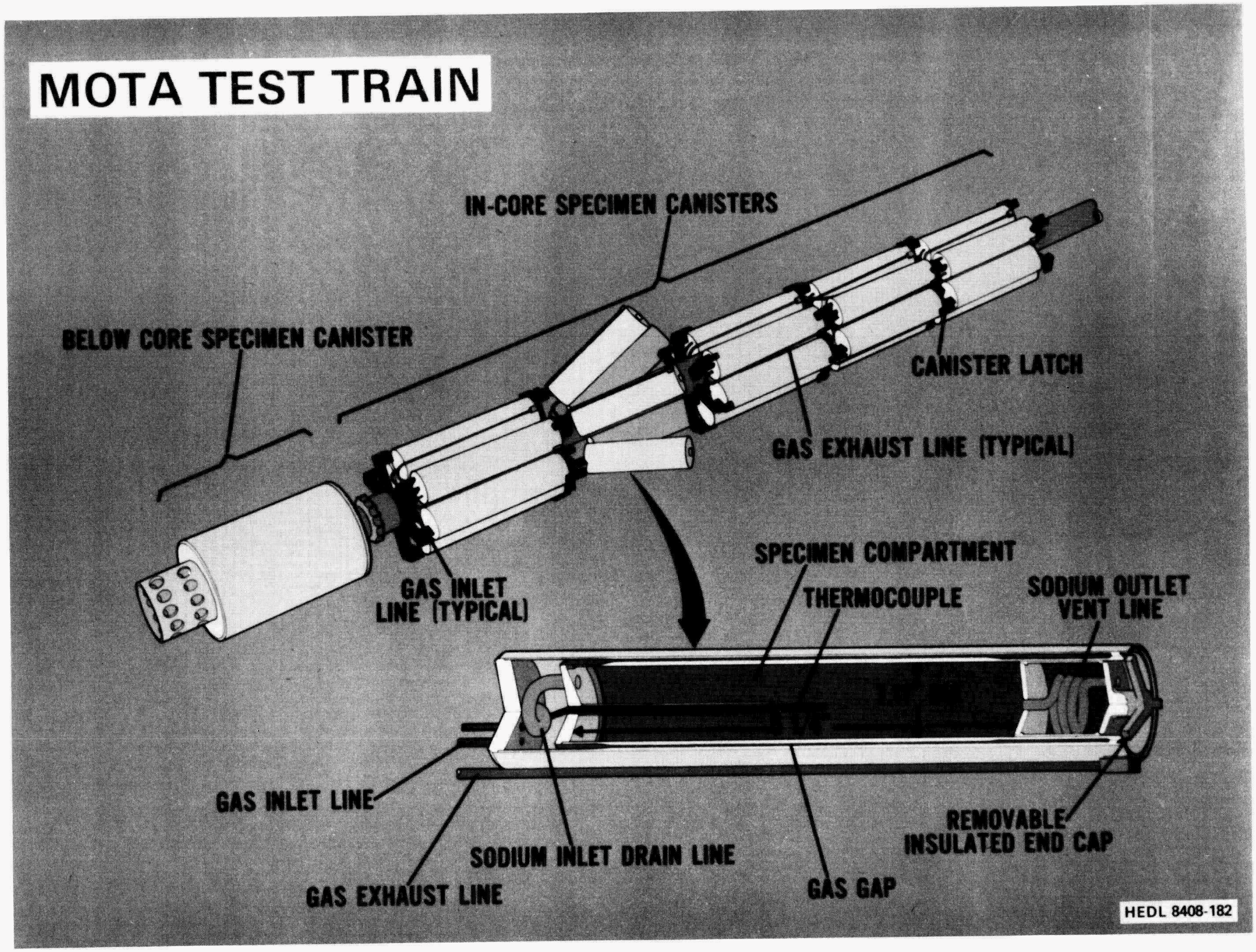
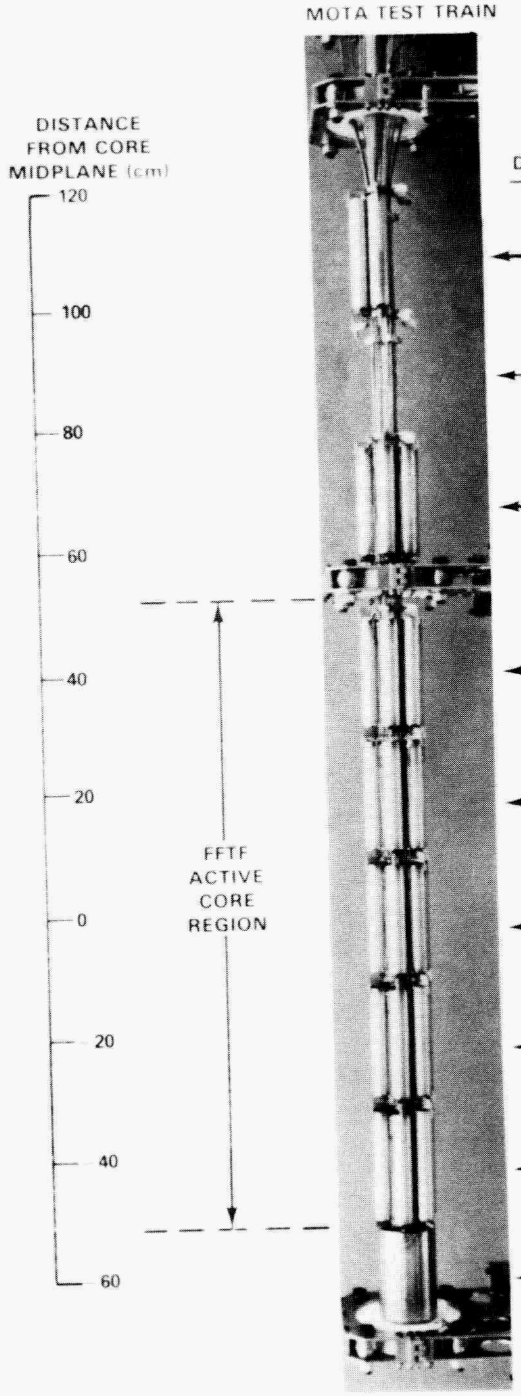

MOTA TEST TRAIN
IN-CORE SPECIMEN CANISTERS
BELOW CORE SPECIMEN CANISTER
CANISTER LATCH
GAS EXHAUST LINE (TYPICAL)
GAS INLET
LINE (TYPICAL)
SPECIMEN COMPARTMENT
THERMOCOUPLE
SODIUM OUTLET
VENT LINE
GAS INLET LINE
REMOVABLE
INSULATED END CAP
SODIUM INLET DRAIN LINE
GAS GAP
GAS EXHAUST LINE
HEDL 8408-182

FIGURE 1

MOTA TEMPERATURES AND FLUENCES



MOTA CANISTER DESIGNATION	AVAILABLE IRRADIATION TEMP. WEEPER CANISTERS	AVAILABLE IRRADIATION TEMP. GAS-GAPPED CANISTERS	MID-CANISTER FLUENCE* (10^{22} n/cm^2) (E > 0.1 MeV)
LEVEL 8	445°C	–	0.045
LEVEL 7	440°C	–	0.17
LEVEL 6	435°C	445-560°C	0.54
LEVEL 5	435°C	490-750°C	3.5
LEVEL 4	430°C	520-750°C	6.3
LEVEL 3	425°C	530-750°C	7.5
LEVEL 2	410°C	515-750°C	6.8
LEVEL 1	390°C	460-750°C	3.9
BELOW CORE	370°C	–	1.2

HEDL 8412 203.5

*CALCULATIONS BASED ON 300 EFPD DURING 14 MONTH IRRADIATION CYCLES (APRIL 86)

FIGURE 2

SP-100 REFRACTORY CANISTER

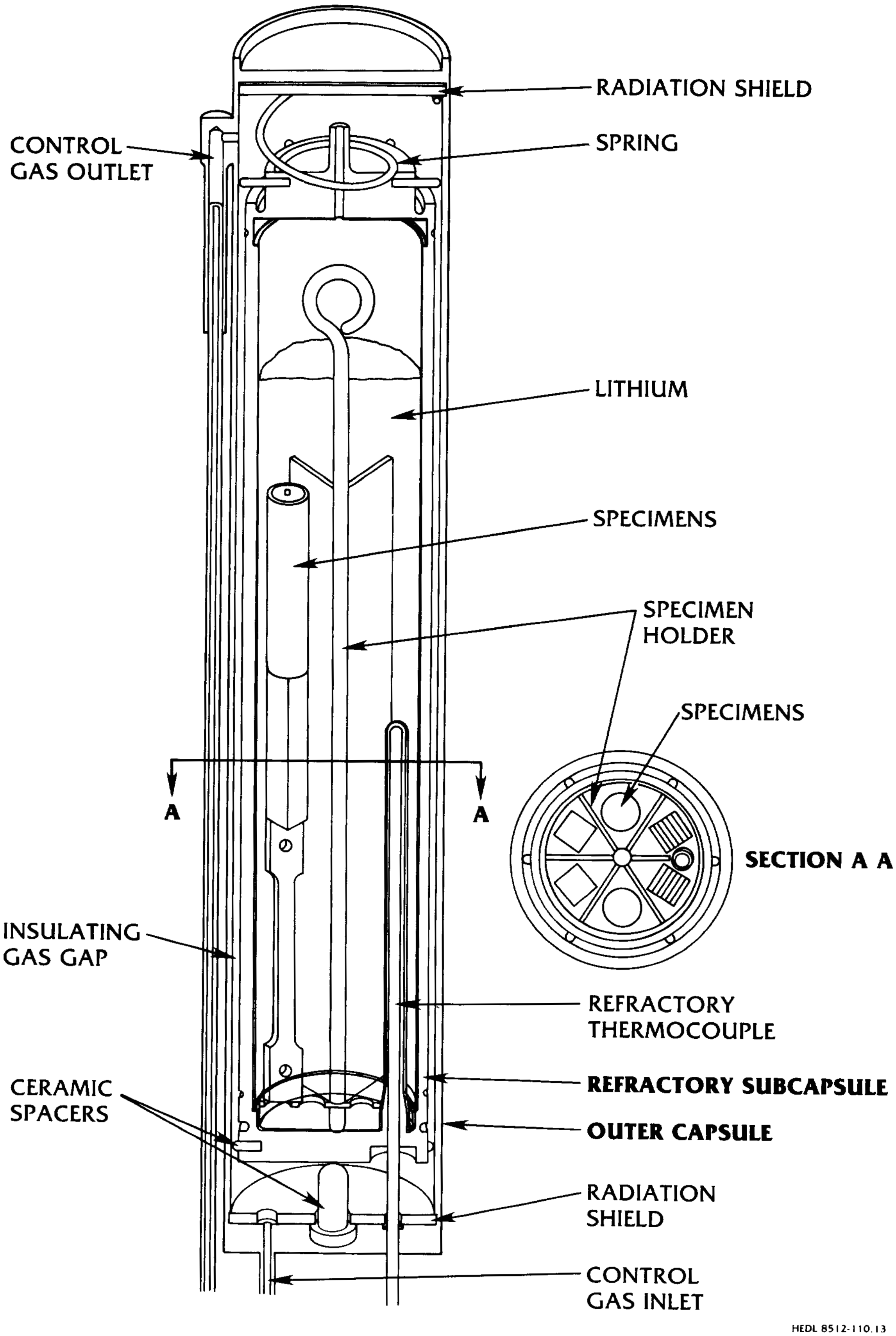


FIGURE 3

FFTF REACTOR SCHEDULE
(October 1986)

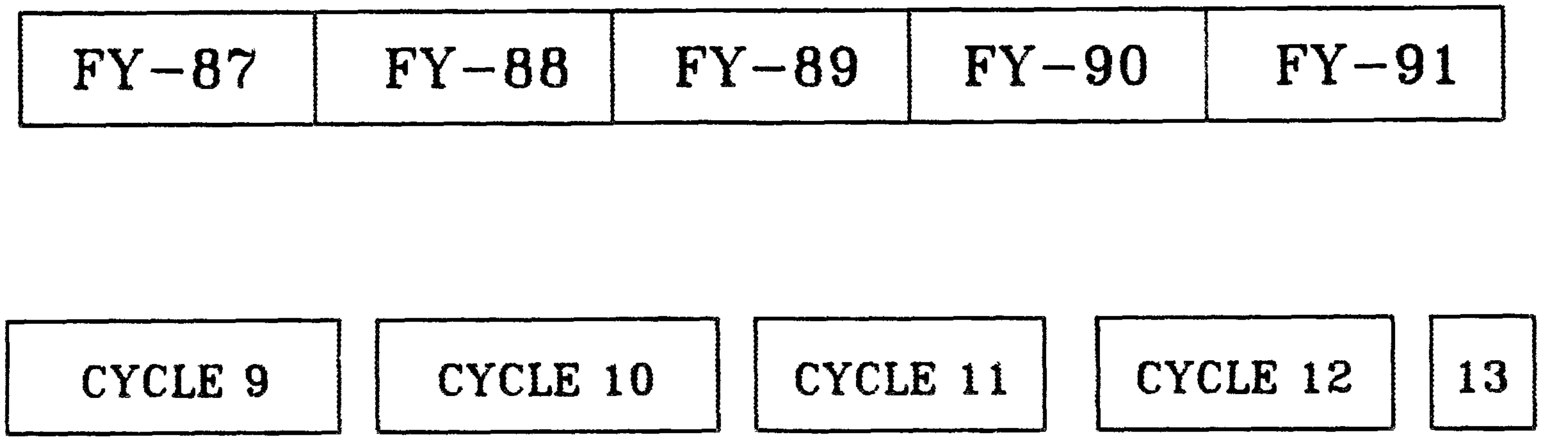


FIGURE 4

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