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HIGH TEMPERATURE MODERATOR PROGRAM

T. Hikido

The purpose of this memorandum is to outline the high temperature hydride moderator program proposed for the Metallurgy Division. The objectives of this program are (1) to provide physical and mechanical property data required by the reactor designers, (2) to develop methods for fabricating moderator assemblies, and (3) to devise and conduct tests to evaluate these assemblies. The requirements in each of these areas and the work proposed to meet them are outlined below. In the conduct of the program, it is planned that full advantage will be taken of the work on metal hydrides being performed by other organizations. These include, General Electric Company; Atomics International; Curtiss-Wright; The Power Plant and Materials Laboratories; WADC; Denver Research Institute for General Electric and WADC; Metal Hydrides; National Bureau of Standards for WADC; and Pratt and Whitney.

I. Physical and Mechanical Properties

The data required by the reactor design engineers and the work planned to supply them are outlined below:

- A. Stress-Strain Curves as function of N_H and temperature at strain rates producing failure in times up to 2 hr.

The inert atmosphere tensile testing apparatus available in the Mechanical Testing Group will be used to conduct tests on the hydrided metal under a hydrogen overpressure. This atmosphere is required to prevent changes in the composition of the specimen during test.

The apparatus has been checked at room temperature on mild steel specimens and Inconel. These tests pointed out some necessary changes which are now being made. These include the substitution of molybdenum pull rods for the stainless steel.

- B. Modulus of Elasticity at Elevated Temperature

In addition to the stress-strain measurements above, some consideration

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will be given to other methods of determining the modulus of elasticity.

The beam deflection apparatus available at WADC, and the sonic equipment at the Ceramics Laboratory will be investigated.

C. Coefficient of Thermal Expansion

The thermal expansion measurements previously made with the equipment available in the Ceramics Laboratory demonstrated the need for maintaining a hydrogen pressure over the specimen to prevent the loss of hydrogen during test. The hydrogen loss results in shrinkage which counteracts the thermal expansion and thus invalidates the tests. Modifications will be made to the present equipment to permit the application of the required hydrogen over pressure. If necessary, new equipment specifically designed for hydrides will be investigated.

D. Thermal Conductivity

The equipment being built by J. P. Page for determining the thermal conductivity of the nickel-molybdenum alloys and other materials is designed for eventual use on hydrides.

E. Heat Capacity

The equipment required to measure heat capacity is available in the Ceramics Laboratory; therefore, these data will be determined there.

II. Fabrication

It is believed that the development of a high temperature moderator can be expedited if a specific design is established as the objective as early as possible. Accordingly, on the basis of presently available metallurgical data and discussions with Mr. A. P. Fraas and his Advanced Design Group, the moderator assembly with the features listed below will be the primary objective. However, other configurations such as flat plates will also be given some consideration. Information such as that obtained on hydriding procedures, cladding, and compatibility studies will be readily adaptable to any configuration.

Proposed Moderator Rod Configuration

- a. Cylindrical Rod, nominally 1 1/2-in. to 1-in. dia x 32-in. long.
- b. Yttrium hydride core.
- c. Duplex of molybdenum and Ni-Mo alloy with total thickness of approximately 0.035-in.
- d. Longitudinal compartmentalization to prevent hydrogen migration.
- e. Structural strength will be provided by clad primarily.

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Probable Operating Conditions

- a. Longitudinal ΔT approximately 350°F.
- b. Internal ΔT , 150 to 350°F.

The following work is proposed in this area.

A. Hydriding Studies

The basic objective of these studies will be to determine the optimum procedure for hydriding metals and to develop these procedures to the point that hydrided metals in various shapes and sizes can be consistently produced to a specified N_H . The correlation between hydrogen content and density and dimensional changes will also be determined.

A hydriding system consisting of furnace, retort, and gas purification train has been constructed and a number of zirconium samples have been hydrided. Improvements indicated by these initial runs are now being made. Equipment capable of achieving the high temperatures required for hydriding yttrium is also available.

B. Cladding Studies

The cladding investigations involve two problems:

1. The bonding of molybdenum to the metal hydrides.
2. The cladding of molybdenum with nickel-molybdenum alloys.

The studies planned on the bonding of molybdenum to the hydrides include the following:

1. Bonding methods

- a. Hot swaging.
- b. Hydrostatic pressure bonding.
- c. Co-extrusion.
- d. Hydriding in situ.
- e. Shrink fitting by heating the molybdenum and pressing in the hydride, followed by hydrostatic pressure bonding, if necessary. Although metallurgical bonding may not be achieved, this method will give a very close mechanical fit which may be adequate because the relative expansion coefficients of Mo and YH_x are favorable.
- f. "Brazing" alloys to promote bonding.
 - (1) Chromium plate
 - (2) Iron plate

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2. The bonds will be evaluated metallographically after thermal cycling and long time exposure to high temperatures.

Two approaches to the problem of cladding molybdenum with a nickel-molybdenum alloy will be investigated. One will be to fabricate molybdenum-NiMo alloy duplex tubing prior to introducing the hydride into the assembly and the second will be the cladding of Mo capsule in which the hydride has been sealed. The methods which will be considered to fabricate duplex tubing will include:

1. Co-extrusion and draw.
2. Swaging.
3. Floturning (Lodge and Shipley Company).
4. Vapor-deposition.
5. Electrophoretic deposition.

The latter two methods will be more in the nature of "last resort" methods to be used if all the others fail.

If the moderators are fabricated by first encapsulating the hydrided metal in molybdenum, the following methods will be tried to clad the capsule with the nickel-molybdenum alloy.

1. Hot swaging.
2. "Sinking".
3. Hydrostatic pressure bonding.
4. Rod rolling.

It will be necessary to study barrier metals to achieve and maintain a good bond between the molybdenum and nickel-molybdenum alloys. The evaluation will again be by metallographic examination after thermal cycling and extended periods at elevated temperature.

Some molybdenum-to-molybdenum bonding studies will be required in connection with end seals and internal barriers. The temperature and amount of work necessary to achieve a bond will be studied. If this proves to be a serious problem the Materials Laboratory, WADC, will be requested to include this specific application in the ultrasonic welding contract they are now negotiating.

C. Investigation of other Hydrogen Diffusion Barriers

Molybdenum has been found to be the best hydrogen diffusion barrier and will therefore be used initially in the fabrication work. However, because of the well known difficulties of fabricating molybdenum, other

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metal barriers will be studied. These barriers have the dual requirement of preventing hydrogen diffusion and being compatible with Yttrium hydride. GEANP reports that Cb, Zr, A and Ho meet the latter requirement. Of these, A would be of most interest.

D. Other Configurations

Since a plate type configuration would offer some advantages from an assembly and cooling standpoint, the fabrication of clad 1/4-in. thick plates will be studied.

III. Evaluation

The evaluation of the moderator rods will be accomplished under conditions simulating those anticipated in reactor operation as closely as possible. Structural and compositional stability will be the properties of most interest. Some of the tests planned at this time are the following:

A. Thermal Cycling and Thermal Gradient

These tests will be used to determine bond integrity and the extent of hydrogen loss and migration. The seesaw apparatus used in corrosion testing may be a useful tool for this test.

B. Mechanical Properties

Creep, stress-rupture, bend, fatigue, and strain-cycling tests will be designed and conducted on clad moderator assemblies.

C. Rate of Hydrogen Loss Through Clad

Apparatus is being set up to measure the rate of hydrogen loss from clad components at elevated temperatures.

D. Corrosion Tests

The corrosion testing program will involve several considerations:

1. Compatibility of molybdenum and nickel-molybdenum alloys in Liquid No. 107. These tests will be run to determine the extent of dissimilar metal corrosion in this system. If these materials prove to be compatible the need for a duplex clad on the hydrided metals would be eliminated and the fabrication may be simplified.
2. The effect of hydrogen on the corrosion of structural alloys by fused salts will be studied in thermal convection loop tests.
3. The effect of corrosion on hydrogen loss through the cladding will also be investigated.
4. A forced convection loop will be used to evaluate an actual moderator rod assembly under reactor flow conditions.

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E. In-pile Tests

An in-pile test program will be conducted in cooperation with the Reactor Design Group and Solid State Division. The Metallurgy Division will help to design the tests, fabricate the test specimens and help to evaluate the results. The first test specimen has been tested in the MTR and has been returned to Oak Ridge for examination.

F. Inspection

Methods will be studied by the Non-Destructive Test Group for the inspection of moderator rod assemblies for bond integrity and uniformity of density and structure. Radiographic, eddy current and ultrasonic methods will be investigated.

IV. Yttrium Metal Production

Inquiries made in the fall of 1956 indicated that it would not be possible to obtain high-purity yttrium metal for an indefinitely long period. At that time a program to produce yttrium metal at the Oak Ridge National Laboratory was initiated in cooperation with the ANP section of Chemistry Division. Initially, the method developed by Ames Laboratory, Iowa State College, which involves the reduction of YF_3 by calcium with additions of Mg and $CaCl_2$, was used. One run was made successfully by this method and yttrium-magnesium alloy was produced.

In March, 1957, a further survey was made of the yttrium metal supply situation. At that time, the picture, both with respect to quantity and purity, still was not satisfactory. It was decided to embark on a research program to develop new methods for producing the yttrium metal. Working with the Chemistry Division, a procedure was developed in which the mixed fluorides YF_3 , MgF_2 and LiF are purified in the molten state, then reduced with Li to produce the yttrium-magnesium alloys. Three successful runs have been made by this procedure. At present the vacuum distillation and vacuum induction melting procedures are being developed.

Proposed future work on the production of high-purity yttrium metal include the following:

- A. Continuation of research on the reduction process to produce the Y-Mg alloy.
 1. Improved methods of loading the fluoride and the lithium.
 2. Investigation of crucible materials, Zn, Ta, Mo.
 3. Time-temperature relationships to improve yields.

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- B. Development of vacuum distillation process.
- C. Vacuum melting of sponge produced
 - 1. Improve vacuum induction melting method.
 - 2. Contact commercial sources for vacuum arc melting.
 - a. Oregon Metallurgical (Working at present for AEC on this problem)
 - b. Allegheny Ludlum
 - c. Metallurgical Research Laboratories, Union Carbide.
- D. Develop large scale production methods.

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