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**REFRACTORY ALLOY DEVELOPMENT FOR THE
ADVANCED SPACE POWER REACTOR PROGRAM**

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March 17, 1969

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REFRACTORY ALLOY DEVELOPMENT FOR THE ADVANCED SPACE POWER REACTOR PROGRAM

Abstract

This report summarizes the work done by the Metallurgy Division for the Advanced Space Power Reactor Program. We characterized the deformation of W, Re, and W-Re alloys at high temperatures and found a basis for developing stronger alloys. A chemical-vapor-deposition process was invented for making W-Re tubing with good mechanical properties. Surface contamination of tungsten particles was found to be the obstacle to the incorpora-

tion of submicron second-phase particles in a tungsten matrix. State-of-the-art tungsten-alloy technology was found wanting but amenable to improvement. Welding or bonding T-111 alloy to W-Re-Mo was unsuccessful. Unusual techniques were created to study corrosion of tungsten alloys by lithium. We established a seven-unit facility for tensile creep testing at 10^{-10} torr at elevated temperatures.

Introduction

This report is a summary of the work undertaken in the Metallurgy Division for the Advanced Space Power Reactor Program. Included are some evaluations and recommendations not presented elsewhere.

The metallurgical program was concerned with two short-range problems: supporting corrosion studies and evaluating existing mechanical property data.

Longer-range objectives—alloy improvement and focusing of fabrication development—were to be based on these preliminary findings. The Advanced Space Power Reactor Program was terminated before the early work could be fully applied to longer-range objectives.

In the selection of reactor structural materials, one must consider mechanical properties, fabricability, and inter-component (structural) compatibilities,

which follow closely in importance the fundamental considerations of fuel-structure and coolant-structure compatibilities. We directed our metallurgical work to these three areas, for which design information was not available.

We began the program having had some experience in chemical vapor deposition, an excellent chemical and metallurgical diagnostic capability, and a well-equipped welding laboratory. However, high-temperature creep equipment consisted of but one ultra-high-vacuum 2400°C furnace, and no fabrication equipment was on hand.

For an early entry into the liquid-metal corrosion phase of the evaluation work, we selected the existing technology of General Electric Company's Nuclear Materials and Propulsion Operation (G.E.-NMPO) for the manufacture of W-Re-Mo

ternary alloy tubing for a pumped loop and corrosion capsules. The longer-range objective was to select and evaluate the coolant compatibility of a stronger W-base alloy. At first the choice was not clear, and tubing was unavailable in stronger materials.

The U. S. Bureau of Mines (USBM) and Wah Chang Corporation were chosen to prepare material for mechanical-property screening studies.

ACCOMPLISHMENTS

We characterized the deformation of W, Re, and W-Re alloys between 1400 and 1900°C, showed the applicability of the data for design purposes, and found a basis for developing stronger alloys.

A chemical-vapor-deposition process was invented and proved for making W-Re tubing with room-temperature mechanical properties at least as good as those of wrought products.

Surface contamination of tungsten particles was found to be the obstacle to

the incorporation of high-melting second-phase submicron particles (such as ThO_2) in a tungsten matrix. A method for producing a dispersion-hardened system was devised but not tested.

State-of-the-art tungsten-alloy technology was evaluated and found wanting but amenable to improvement.

Welding or fusion bonding of T-111 alloy to W-Re-Mo was unsuccessful. By analogy with previous findings with Ta-Mo alloy single crystals, we expect reliable metallurgical joints directly between Group V and Group VI metals to be difficult to attain.

Unusual techniques were created for assembling and metallurgically analyzing lithium tungsten-alloy corrosion experiments.

During this program we established a seven-unit facility for tensile creep testing at 10^{-10} torr at temperatures up to 2600°C.

Elaboration of the above accomplishments, as well as recommendations for future work, are discussed in the following sections.

Mechanical Properties and Physical Metallurgy

Reactor-loop structural materials and fuel cladding must resist gross plastic deformation by creep at high operating temperatures. Structural elements, in addition, must withstand the acceleration loads of launch and the thermal stresses of startup when their mean temperatures are below or near the ductile-brittle transition temperature of tungsten. It is well known that rhenium imparts room-temperature toughness to tungsten. In fact, the body-centered-cubic W-25 at. % Re solid-solution alloy, prepared by

chemical-vapor deposition at LRL, showed a 75 percent reduction in area in a room-temperature tensile test. The beneficial effect of Re on W at low temperature and the uncertainty of the influence of Re on the strength of W at high temperature were the motivations for including W-Re alloys in our deformation studies.

All our creep tests were performed between 1400 and 1900°C at stresses below 10,000 psi.^{1,2} Some of the steady-state tensile creep data for the metals tested are compared with the compressive

strength of uranium nitride³ in Table I. All these metals show promise as cladding materials that could resist diametrical fuel swelling.

Tungsten and all of the alloys had steady-state creep rates ($\dot{\epsilon}$) that could be

Table I. Steady-state creep rates measured at 1600°C and 2500 psi.

UN	$1 \times 10^{-3}/\text{hr}$
W	$7 \times 10^{-5}/\text{hr}^a$
W-25 Re	$5 \times 10^{-5}/\text{hr}$
W-5 Re	$6 \times 10^{-6}/\text{hr}$
Re	$8 \times 10^{-5}/\text{hr}$

^a Not including primary strain of 1 to 2 percent, which occurs rapidly after application of load.

characterized by the expression $\dot{\epsilon} \approx \left(\frac{\sigma}{E}\right)^n \exp \frac{-100}{RT}$, where σ is the applied stress, E the modulus of elasticity at test temperature, R the universal gas constant, and T the absolute temperature. For W and W-5 Re, the stress exponent n was 5.8; for W-25 Re and W-30 Re, n was 3.8. However, we observed a change in mechanism when fine-grained W-25 Re was tested at lower strain rates. This change in mechanism illustrates the necessity of performing long-term tests for reactor design data. Extrapolation of the present data to lower strain rates can most conservatively be done by limiting n to 1 at applied stresses less than 2500 psi (Fig. 1). However, it hasn't been demonstrated that these higher stress exponents do not hold at lower stresses in most cases.

Rhenium additions significantly altered the deformation behavior of W at elevated temperature. At stresses in excess of 2200 psi, large additions strengthened W.

Fig. 1. Strain rate ($\dot{\epsilon}$) vs stress (σ) data for tungsten alloys—extrapolated to lower strain rates.

Tungsten-5 Re alloy, however, was stronger than W, W-25 Re, or W-30 Re at all stresses less than 10,000 psi between 1400 and 1900°C. In all concentrations, Re eliminated the 1-2 percent primary creep strain found in W; i.e., steady-state creep rates were immediately established when the load was applied to the alloys.

In general, thermomechanical history and variations in minor-impurity contents did not influence the creep strength of W or W-Re alloys.¹ However, W and W-Re with grain sizes greater than 50 μ were stronger than finer-grained material at stresses below 3500 psi; i.e., they crept more slowly for a given stress. Also, evidence was obtained that the interplay of second-phase particles and plastic deformation could be a basis for strengthening W and the W-Re alloys.

Tungsten-30 Re alloys contained a grain-boundary precipitate of the very hard (approximately 1600 DPH) sigma inter-metallic phase. Although the transmission electron microscopy studies revealed dislocation-sigma particle interactions

within the grain, the interactions were not extensive enough to decrease the creep rate; the mobility of dislocations was not appreciably influenced. Future studies should include attempts to nucleate a finer sigma dispersion within the matrix grains.

Equilibrium dislocation densities were proportional to the 2.3 power of the stress for tungsten and the alloys. Tungsten alone developed subgrains of a size inversely proportional to the applied stress and independent of temperature; the primary creep strain mentioned previously was attributed to the formation of this substructure. Figure 2 shows two creep curves at 1600°C for tungsten. For the test at 3500 psi, there was a steady-state rate of about $7 \times 10^{-4} \text{ hr}^{-1}$. For the other test, steady state was established at 6000 psi and then the load was reduced to 3500 psi, where the creep rate dropped to about $7 \times 10^{-5} \text{ hr}^{-1}$ but gradually increased to the value expected at 3500 psi. If the finer substructure established at a higher stress could be stabilized at a lower stress by hard dispersed second-phase particles, appreciable strengthening would be obtained.

Rhenium also has a high melting point (Re: 3180°C; W: 3410°C) and high elastic modulus (Re: $66.7 \times 10^6 \text{ psi}$; W: $50 \times 10^6 \text{ psi}$). Because it has an hcp structure it was important to compare rhenium with bcc tungsten in creep. Figure 3 summarizes the test results. Between 1500 and 2200°C, the

Fig. 2. Creep curves for tungsten at 1600°C.

steady-state creep rate of Re depended on stress to the 3.4 power.⁴ The apparent activation energy for creep was 60 kcal/mole, in contrast to 100 for tungsten and the alloys. There was no primary creep for the conditions of temperature, stress (1,500-10,000 psi), or grain size studied. Anomalous in Re, dislocation densities in annealed or annealed-and-crept specimens were $10^9/\text{cm}^2$, invariant of stress or temperature. Since Re has limited slip systems, dispersion hardening could prove exceptionally effective in increasing its creep resistance.

Fig. 3. Comparison of creep data for tungsten and rhenium.

Fabrication Development

CHEMICAL VAPOR DEPOSITION

Hydrogen reduction of WF_6 and coreduction of WF_6 with ReF_6 were used to produce mechanically sound tubing for liquid-metal containment; e.g., cladding or heat pipes. Reaction parameter studies were

done on short lengths to find ways of making a range of strong alloy compositions at useful rates.

Later, we built an apparatus⁵ to make long tubes (see Fig. 4). We used it to make 1-ft lengths of W-25 Re tubing 1/2 in. in diameter by 0.040-in. wall. Longer

Fig. 4. Chemical-vapor-deposition apparatus for making long tubes.

lengths, other sizes, thicker walls, and other compositions could be produced without significantly changing the basic design.

In operation, premixed metal fluorides are introduced with H_2 into the reaction zone, and the gas mixture flows vertically across a 4-in. length of rotating molybdenum mandrel. Metal deposits on the heated mandrel, which is translated through the heated zone at a rate that depends on the thickness desired and on the required reaction parameters (pressure, temperature, mole ratios, flow rates, etc.). For the W-25 Re alloy, the deposition rate was approximately 0.0003 in./min. The inside surface of the tubing was a replica of the outside surface of the sacrificial mandrel. Thus, the as-plated tubing required finishing on the outside diameter only. Chemical

homogeneity in the W-Re deposits was closely controlled with the aid of an induction coil designed to minimize temperature gradients in the reaction zone.

Future work could be directed toward an apparatus for plating large cylindrical shells (say, 18 in. diam $\times$ 36 in. long) for reactor pressure vessels. Fine-grained, sound, and homogeneous microstructures have been deposited at a rate of 0.00075 in./min. Further studies should find conditions that produce good deposits at a rate of 0.001 in./min.

Sound deposits of alloys containing as much as 30 percent Re were made in short lengths.⁶ Figure 5 summarizes the room-temperature mechanical properties of these various compositions. Mechanical properties of many of the alloy deposits made

Fig. 5. Tensile strength of CVD W and W-Re alloys.

under optimum conditions⁷ are as good as those found in wrought material originating as an arc melt or as a powder-metallurgy billet. An electron-beam butt fusion weld between two CVD tubes of W-25 Re was free of porosity. Elevated-temperature properties have yet to be compared with those of the materials discussed under mechanical properties and physical metallurgy. Those materials were all rolled from powder-metallurgy billets.

Chemical-vapor deposits typically have coarse, columnar microstructures that are frequently porous and usually mechanically weak. We have used mechanical brushing during plating or deposition of a metastable phase to control the microstructure of all compositions, thereby obtaining good room-temperature mechanical properties.

Mechanical brushing during deposition of alloys containing up to 18 percent Re produces frequent nucleation of new crystals and results in a controlled fine grain size.⁷

Achieving a fine-grained deposit in alloys containing more than 18 percent Re depends mainly upon another phenomenon. During deposition of the richer compositions, metastable federite (A-15 type phase) forms and coexists with the body-centered-cubic W-Re solid solution. With the proper choice of reaction parameters, the deposit will contain a fine dispersion of this phase. During subsequent annealing, the dispersed metastable phase transforms to the equilibrium structure, and the resulting product is a fine-grained, equiaxed solid solution.

Deposits with more than 28 percent Re contain a fine dispersion of sigma. Chemical-vapor deposition may be capable of producing a more optimum distribution of sigma than is obtained by hot working the W-Re powder-metallurgy billet. Thus the process, which offers a way to improve high-temperature properties, should be explored.

POWDER METALLURGY

A suitable dispersion of insoluble, high-melting particles could stabilize the fine subgrain sizes that develop² when tungsten is crept at high stresses (recall Fig. 2). Interparticle separations should be less than 10 μ , and the dispersoids themselves should not represent a large fraction of the composite. A desirable dispersoid would be 2-5 vol% submicron ThO₂ particles.

Historically, attempts to incorporate ThO₂ by blending submicron powders and sintering have resulted in coarsening of the ThO₂, with the coalescence of the dispersoid occurring at the tungsten grain boundary. In our work we discovered⁸ that densification in wet hydrogen, attended by thoria coarsening, proceeded at a much lower temperature (approx 1100°C) than the 2500°C temperature normally used for sintering these composites. A tentative explanation for the activated sintering involves the formation of "liquid" interparticle films associated with an interaction between thoria and a tungsten oxide derived from oxygen initially chemisorbed on the particle surfaces. When large volumes (30 percent) ThO₂ were sintered with tungsten in moist hydrogen at 1335°C, all the ThO₂ apparently was not consumed by reaction with the tungsten oxide, and the

unreacted ThO₂ was present within the tungsten grains as particles smaller than 0.1 μ .

These observations suggest a powder method for preparing tungsten strengthened with ThO₂. The product of a first sintering process could be coarse-ground to liberate the coarse ThO₂ from the weakened grain boundaries. After separation of the large ThO₂ particles from the W-fine ThO₂ composite on the basis of density, the composite powder could be consolidated by hot pressing and/or sintering without further agglomeration, because the ThO₂ inclusions would not be available to the new composite-particle surfaces.

Powder densification without additives that may detrimentally influence mechanical properties requires pressure at a moderately high temperature or pressureless sintering at very high temperatures. A consolidation survey⁹ with small-scale apparatus has shown that 98+ percent densification can be achieved in a few seconds at 1 kbar and 900°C. Pressures up to 30 kbar and temperatures to 1400°C were included in the survey. The Laboratory has a newly installed hot isostatic press that permits the consolidation of a billet up to 8 in. diam $\times$ 16 in. long with 2-kbar gas pressure at 1600°C. We expect that our laboratory findings could be scaled up to produce extrusion billets if necessary.

STATE-OF-THE-ART ALLOY

G. E. -NMPO technology for making W-25 Re-30 Mo and W-30 Re-30 Mo alloy tubing was the basis of our selection of alloys for initial lithium corrosion experiments. Tooling was designed and built for electron-beam welding G. E.-produced

components at LRL to make a pumped loop. Machine parameters were established for butt-welding the tubing with our Hamilton Standard electron-beam welder.¹⁰

Satisfactory full-penetration welds were obtained in the G. E. powder-metallurgy product. However, weld-line porosity after a single-fusion pass was substantially greater than in arc-melt alloy obtained from USBM and G. E. Multiple-pass welding reduced the porosity. We finally settled on a two-pass technique to minimize porosity without severe upsetting (dimensional distortion). From the weldability standpoint, the tubing was satisfactory.

The time-consuming G. E. process for powder and green-compact purification (prior to sintering, hot extrusion, and cold drawing) reportedly eliminated evolution of gases from the finished base metal during welding. It didn't. And, in fact, the need for the expensive process should be re-examined. There are some advantages to the powder-metallurgy process, and therefore this comparison of weldability is not meant to indicate a preference for the arc-melt-origin tubing. Process selection will depend on specific design objectives.

Ternary alloys are at least a factor of 10 weaker in creep near 1400°C than the binary alloys. However, they are apparently more amenable to conventional tube-manufacturing techniques. Welded and unwelded tubing of the ternary alloys is now being evaluated by 1500-psi longitudinal tensile-creep proof tests at 1500°C. The creep tests will be continued for several thousand hours.

Low-temperature testing of ternary alloys consisted of comparing as-received and fusion butt-welded tubing in tension between room temperature and 400°C.

Fusion-welded specimens were as strong and tough as coupons machined from as-received tubing.¹¹ Analysis of the test data (Fig. 6) suggests that the G.E.-NMPO drawing process is done too cold (200°C) and should be performed at 400°C, where the ductility is higher. In general, the whole cold-working part of the G. E. process could be substantially improved. Strain rates and reductions should be studied in addition to temperature.

The inability of G. E.-NMPO to produce enough sound tubing in time prevented construction of the loop. About 90 percent of the tubing received at the Laboratory was rejected for loop use because it had cracks that extended from the inside surface to as much as half way through the wall. Nondestructive inspection methods were developed at LRL that were sensitive to very shallow flaws as well as to these large ones that had escaped detection at G. E.-NMPO. Standards and techniques were established for in-process inspection at G. E. with the hope of saving time and money in obtaining the balance of the material required for the loop.

Attesting to the low-temperature toughness of these alloys were the deep cracks that had widely opened (large included angle) without propagating through the 0.040-in. wall. The wide openings were indicative of gradual propagation during several of the multiple-drawing operations used to reduce the wall 5 percent per pass. Each of these cold-drawing operations required a new mandrel. It is doubtful that the existing G. E. -NMPO process could be applied to the stronger W-Re binary alloys. As stated before, W-Re alloy tubing can be made very easily by the LRL chemical-vapor-

deposition process. Compositional control of the three-component-alloys would be

very difficult, however, by chemical-vapor deposition.

Fig. 6. Temperature dependence of engineering tensile properties of W-25 at. % Re-30 at. % Mo, 0.5-in.-o.d. $\times$ 0.040-in.-wall tubing supplied by G. E.-NMPO.

Interpart Compatibility

The coupling of a tantalum-alloy rankine power-conversion system to a tungsten-alloy reactor may require joints between these dissimilar alloys. Fusion welds were tried between W-25 Re-30 Mo and Ta-8 wt% W-2 wt% Hf (T-111).¹¹ Direct welds were tried, as well as joints with Mo or Mo-50 Re transition consumable

inserts in the fusion zone. All joints cracked.

Analyses of the fusion zones and fracture surfaces suggest a fundamental problem. Although no second phases are formed between the Group V (Nb, Ta) and Group VI (W, Mo) metals, Van Torne and Thomas¹² report solute clustering on

the (100) plane in Mo-Ta single crystals and a lowered cleavage stress. Enough corroborating evidence has been collected in our work to say that there is little hope of getting sound, reliable fusion welds directly between components of the two chemical groups. Diffusion bonds between W-25 Re-30 Mo and T-111 were made in 30 min at 1600°C and 10,000 psi. They also were fragile and cracked at the interfaces during test-specimen machining. Brazing with a third group metal would depend upon the degree of interdiffusion at reactor operating temperatures. Such joints were not tried.

Interface compatibility was the only compatibility problem considered (other than liquid metal by others) during the program. Potential problem areas in

multiple-component systems, in addition to the example above, are the transfer of, say, W onto Ta or Ta onto W through a common liquid-metal stream or the transfer of nitrogen from a dispersion-hardened Group VI alloy to a Group V alloy. One could speculate on a long, long list of difficulties.

Although all the engineering solutions for the construction of a chemically simple power system are not in hand, in our opinion the cost of fabrication development and material characterization for a system composed of many chemical components is unjustifiable, especially when the complications of inter-component compatibility are considered. Further, the performance limitation on either temperature or redundancy cannot be justified.

Liquid-Metal Corrosion—Metallographic Support

The results and significance of the lithium corrosion work and recommendations for future work are more properly discussed by others.¹³ A brief resume of the metallurgical support to the program is included here to highlight the techniques that were devised at the Laboratory for examination of corrosion samples.

The corrosion damage sustained by a W-25 Re-30 Mo alloy after 1000-hr, high-temperature exposure to flowing lithium was examined metallographically.¹⁴ A test strip was prepared by electropolishing the surface at hot and cold regions prior to exposure. After exposure to liquid metal, polished areas were analyzed by the electron-beam microprobe. Optical

micrographs and electron micrographs of replicas were taken from the same areas. Figure 7 summarizes the results. Molybdenum was transported to the colder end of the strip.

Interpretation of the micrographs led to the conclusion about the topology near grain boundaries illustrated by Fig. 8. All of the topography of the sample was developed in the exposure. Microprobe analyses show that the surface roughening was at least in part due to long-range transport, but local redistributions over distances of the order of a grain size may also be responsible for some of the topography. At this point we cannot be certain of the details of the

Fig. 7. Microprobe analysis and metallographic examination of W-25 Re-30 Mo alloy after exposure to flowing lithium. The four numbers designate the spots electropolished before exposure.

Fig. 8. Schematic diagram of groove shapes developed at the surface of a W-25 Re-30 Mo plate exposed to flowing lithium.

removal-precipitation process in this single experiment, but we feel that we have a powerful set of tools for corrosion studies.

More conventional examination of the container itself (micrography of sections through the wall) revealed 0.001-in.-deep intrusions into grain boundaries at the hot end. Such intrusions, depending on their rate of development beyond 1000 hr and their effect on mechanical properties, could severely limit the use of W-Re-Mo alloys for the containment of dynamic liquid lithium above 1400°C.

No adverse effects of the lithium environment on the low-temperature mechanical properties of the containment alloy were observed after the exposure.

Summary

We conclude that tubing and even large shells of alloys such as W-5 Re and W-25 Re can be made by chemical-vapor deposition. In addition, chemical-vapor deposition should prove useful for welding dispersion-hardened alloys that are not amenable to fusion welding. The tube manufacturing process itself is a continuous welding process.

All the tungsten-base alloys and the W-5 Re alloy, in particular, are in an underdeveloped state relative to T-111 tantalum-base alloy. Nevertheless, single-phase W-5 Re is as strong as T-111 and can potentially be improved by the same strengthening mechanism that has been used for T-111; namely, the use of second-phase particles to decrease dislocation mobility.

With respect to microstructural features, crept materials fall into three categories:

- 1) bcc tungsten, which develops subgrains and a stress-dependent dislocation density;
- 2) bcc tungsten-rich rhenium alloys, which do not develop subgrains but do develop a stress-dependent dislocation density; and
- 3) hcp rhenium, which develops neither subgrains nor a dislocation density characteristic of the creep conditions.

To complete the survey of the W-Re alloy system and select materials for dispersion hardening, rhenium-rich (hcp-matrix) alloys should be tested. Extensive alloy development, however, should only proceed in concert with liquid-metal,

metal vapor-liquid, and fuel compatibility work.

We believe an entire reactor-rankine power conversion system could be made from alloys such as Mo-Re, W-Mo-Re, W-Re, or Re. If it were advantageous for fabricability to use the weaker molybdenum-

containing alloys in the colder parts of the system, the G. E. technology could be readily improved. Technological development of the W-Re alloys would be applicable to a system using thermionic conversion from a higher-temperature source than appears useful for rankine conversion.

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