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PE-16 Swell - 42
- Tensile - 48
Mo Band DBTT - 89

Alloy Development for Irradiation Performance

Quarterly Progress Report

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An adaptation of an electrical potential technique to measure crack growth on miniature center-cracked specimens has been shown to produce reliable, accurate results. Measurement of crack growth rate (da/dN) using this technique is at least as accurate as visual methods. As developed, the technique is conducive to high-volume postirradiation fatigue crack growth testing.

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We calculated the effects of C-3 core piece composition and added fuel loaded around the C-3 position in the ORR. Adding 20 g fuel in each of the positions B-3, C-4, and D-3 reduces the thermal flux by about 4% and increases the dpa by about 6%. Additional calculations are contemplated for larger fuel loadings. A conceptual design of the experimental vehicle has been completed and the core pieces required for the first two stages of spectral tailoring are being fabricated. The composition of the experimental capsule differs from that used in the initial scoping calculations. As soon as final detailed information on the composition becomes available, the neutron flux and dpa will be recateutated. Calculations are in progress to obtain cross sections for neutron absorbers to be used in the latter stages of spectral tailoring to reduce the thermal flux.

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The fatigue testing system for in-cell testing has been completed and is currently being operated for testing irradiated specimens under cyclic fatigue loading. Excellent performance has been demonstrated for a few initial in-cell tests on two irradiated and two unirradiated 20%-cold-worked type 316 stainless steel specimens at 430°C in high vacuum.

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Miniature tensile specimens of types 316 and 316 + 0.23% Ti (TiM 316) stainless steel in the annealed and 20%-cold-worked conditions were irradiated in HFIR

to displacement damage levels of 5-17 dpa and 200–1000 at. ppm He. The specimens were tested near the irradiation temperatures. Reduction of area and true fracture stress were determined, and scanning electron microscope (SEMI fractographs were made to study the fracture mechanism and the effect of titanium on fracture. Type 316 stainless steel showed an unusual brittle behavior at 450°C. Associated with this behavior was a cavity structure visible on the fracture surface. The cavities are tentatively believed to be associated with helium.

3.2 Status of Path A Prime Candidate Alloy Procurement and Fabrication (ORNL) 40

A 1.4-Mg heat of Fe–16 Ni–14 Cr–2 Mo–0.3 Ti–2 Mn–0.5 Si–0.05 C (wt %) was double vacuum melted. So far 420 kg of 100-nnn bar has been received and smaller bar and 13-mm plate have been rolled.

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4.2 Mechanical Properties of Nimonic PE–16 Following Irradiation in HFIR (ORNL), ... 48

Irradiation of solution treated and aged Nimonic PE-16 to produce 3 to 24 dpa and 180 to 4660 at. ppm He produced little or no change in the ultimate tensile strength of samples irradiated and tested near 55 or 500°C. In contrast, the yield stress was raised well above the control value by irradiation at either temperature. Samples irradiated at 55°C and then tested at higher temperatures showed a local minimum in strength change relative to control values and a local maximum in ductility at 600°C. At 600°C test temperature the failure mode was stilt transgranular. Tests at 650 or 700°C following the 55°C irradiation resulted in low elongation to failure. The fracture mode at 700°C was predominately intergranular separation. In samples irradiated and tested at 500°C the elongation remained above 5% for irradiation producing up to 8 dpa and 1000 at. ppm He but dropped below 2% for 11.5 dpa and 1700 at. ppm He or greater.

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Three vanadium alloy and two niobium alloy compositions are being prepared for consumable arc melting and processing to plate, sheet, and rod for the Fusion Materials Stockpile. These are the Path C (V,Nb) Scoping Alloys selected for initial evaluations as candidate fusion reactor structural materials. First-melt electrodes of the niobium alloys have been prepared and are ready for melting. Preparation of the vanadium alloy electrodes has been delayed due to difficulties associated with breakdown of the as-received 3.8 cm (1.5 inch) diameter vanadium rods. The nitrogen level of the vanadium is much higher than reported by the vendor; this has made it impossible to cold-roll the rod to electrode strip as was originally planned. The vanadium rods are being cold-swaged to a size where they can be further processed to thin strip for preparation of the consumable arc-melt electrodes.

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The microstructure of Ti-62425 was studied by transmission electron microscopy (TEM) after irradiation in EBR-II to a fluence of 3.7×10^{21} neutrons/cm^2 ($E > 0.1$ MeV) at 400°C. The overall phase distribution in the alloy, equiaxed primary alpha with a small amount of transformed beta, was unaffected by this irradiation exposure. Electron diffraction patterns indicated the probable presence of the alpha-2, $Ti_3(Al,Sn)$ but it could not be determined if this phase precipitated during heat treatment or during irradiation. The principal. irradiation-induced microstructural feature was a dense distribution of small dislocation loops in the primary alpha grains. The individual alpha plates in the transformed beta were very fine, as before irradiation, but they appeared to be somewhat irregularly shaped, possibly due to interference from the contrast of irradiation-induced lattice defects.

5.3 Comparison of Titanium Alloy Ti-6Al-4V and Ti-5621s Microstructures After EBR-II Irradiation (McDonnell Douglas) 72

Microstructural analysis was performed on two types of titanium alloys, the a t β alloy Ti-6Al-4V in the mill anneal and duplex anneal condition and the near alpha alloy Ti-5621S in the duplex anneal condition after neutron irradiation. The irradiations were made in ROW VII of EBR-II to a fluence of 3.4 × 10^{21} n/cm^2 (E_n > 0.1 MeV) at 450°C. This fluence results in roughly 1.5 dpa. Analysis of the Ti-6Al-4V revealed that both conditions of Ti-6Al-4V contained a fine precipitate of what is believed to be beta phase and a high density of dislocation loops and debris. The precipitates are essentially ellipsoidal in shape and are roughly 100–200 Å in size. The precipitates appear to follow the Burgers relationships and were found to occur at low angle grain boundaries and along dislocations which were produced during the heat treatment. These precipitates appear to be unique to irradiated titanium and have not been observed as a result of long term thermal exposure. The Ti-5621S alloy showed no evidence of this precipitate. The primary defect structure of the Ti-5621s was extensive dislocation loops and c t a type of dislocations.

5.4 Effect of Reactor Chamber Environment on Niobium Alloys (McDonnell Douglas) 85

Niobium, like the other group Vb refractory metals is sensitive to contamination from interstitial elements such as oxygen, nitrogen, carbon, and hydrogen. These elements, if present in sufficient quantities can significantly alter the tensile properties, reduce ductility, and raise the ductile-to-brittle transition temperature to well above room temperature. Therefore, particular attention must be paid to the operating environment of the refractory metals and the effects of that environment on mechanical properties.

Postulated reactor chamber atmospheres of inert gas or low quality vacuum appear to be unacceptable for niobium unless the residual gas pressures are extremely low. For example for operating temperatures up to 600°C, the solubility of carbon and nitrogen is low, that of oxygen moderate, and hydrogen very large. With the exception of hydrogen, increasing temperature raises the solid solubility of these elements. For the typical operating temperatures and pressures likely to be experiences by the refractory metal piping (i.e., ~600°C and a chamber pressure of 10^{-3} torr) the equilibrium oxygen content of the niobium would be roughly 0.1 wt % which can cause embrittlement. Therefore, in order to

maintain an equilibrium concentration below 0.04 wt % requires a vacuum on the order of 10^{-9} torr and a low leak rate.

5.5 The Ductility in Bending of Molybdenum Alloys Irradiated Between 425 and 1000°C (ORNL) 89

Samples of Mo, Mo–0.5% Ti, and TZM (Mo–0.5% Ti–0.09% Zr), initially fully recrystallized, were irradiated in the EBR-II to fluences of 2.5×10^{26} n/m^2 (>0.1 MeV) in the range 425 to 1000°C. Rectangular coupons 3 × 2.8 × 0.4 mm were electropolished and then tested in vacuum in slow three-point bending from 22 to 650°C. The DBTT was bracketed by establishing the shift from failure in bending to full 45" bend under load.

Major findings were: (1) The three materials show similar behavior. (2) The DBTT is a strong function of the irradiation temperature, with the greatest degradation of behavior produced at 585°C. (3) Brittle failures in molybdenum contain a large surface fraction of exposed grain boundaries, from grain boundary separation. The balance of the surface is covered by cleavage facets. In the two alloys, the cleavage facets are the predominant surface feature, but a component of grain boundary separation is still present. (4) The fraction of grain boundary separation increases as the test temperature approaches the DBTT, but ductile tearing is observed above the DBTT.

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Weight loss data for a type 316 stainless steel loop that circulated lithium with 500 wt ppm N showed no major difference from a similar loop with purer lithium when differences in flow velocity were taken into account. Further data on weight loss versus exposure time to flowing lithium are reported. Steady-state corrosion rates generally range from 7 to 21 mg/m^2 h between 540 and 600°C. A simplified analysis of the steady-state corrosion rate as a function of temperature indicates that the corrosion process is controlled by liquid-phase diffusion.

8.3 Hydrogen Permeation Characteristics of Titanium-Base Alloys (ANL) 128

The hydrogen permeation characteristics of the alloy Ti-6Al-4V have been evaluated. This alloy was found to have a permeability nearly 1000 times greater than that of conventional 300-series stainless steels. The dependence of the permeation rate on hydrogen pressure, P, was observed to vary over the range from $P^{0.4}$ to $P^{1.0}$. This result was construed as evidence for a permeation process that is at least partially influenced by surface impurity effects. The surface condition and mechanical integrity of the Ti-6Al-4V membrane appeared to be unaffected by ∿1400 hours of hydrogen infiltration under typical power-reactor plasma-chumber conditions with respect to temperature, gas pressure, and gas composition. A nitride coating (applied by ion-implantation methods) on a second Ti-6Al-4V sample does not appear to have caused a significant reduction in the gross permeability observed for the initial sample.