



Alloy Development for Irradiation Performance

Semiannual Progress Report
For Period Ending September 30, 1984

U.S. Department of Energy
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Disks of EA (in several pretreatment conditions) and several heats of cold-worked (CW) type 316 and D9 type austenitic stainless steels have been irradiated in HFIR at 300, 504, and 600°C to fluences producing about 10 to 44 dpa and 450 to 3600 at. ppm He. These samples are being reirradiated in the Materials Open Test Assembly (MOTA) in FFTF at 500 and 600°C, together (side by side) with previously unirradiated disks of exactly the same materials, to greater than 100 dpa. These samples, many of which have either very fine helium cluster or helium bubble distributions after HFIR irradiation, are intended to test the possibility and magnitude of a helium-induced extension of the initial low-swelling transient regime relative to the void swelling behavior normally found during FFTF irradiation. Further, these samples will reveal the microstructural stability or evolution differences that correlate with such helium effects.

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Vanadium specimens of V-15Cr-5Ti, VANSTAR-7, V-3Ti-1Si, and V-20Ti are being irradiated in the FFTF-MOTA experiment. Miniature sheet tensile specimens and transmission electron microscopy (TEM) disks were preimplanted with ³He to levels up to 480 at. ppm using the tritium trick and encapsulated in TZM capsules containing ⁷Li. The specimens will be irradiated to damage levels up to 165 dpa with irradiation temperatures of 420, 520, and 600°C. All of the capsules at 600°C underwent a temperature excursion during their initial cycle and had to be replaced.

2.11	Irradiation Experiments for the U.S./Japan Collaborative Testing Program in HFIR and ORR (Oak Ridge National Laboratory)	66
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The assembly of all eight capsules, HFIR JP-1 through JP-8, has been completed, and they are now being irradiated. The parts fabrication assembly and flow testing of the prototype ORR capsules for 60°C and 200°C MFE6-J were completed and the capsule was inserted in the ORR. Performance was satisfactory in all respects.

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Specimens of three vanadium alloys were implanted with different levels of ³He using the tritium trick and subsequently tensile tested at elevated temperatures. The V-15Cr-5Ti and V-3Ti-1Si specimens were embrittled by ³He at a level of 150 at. ppm while the VANSTAR-7 specimens were not. The embrittlement appeared to be caused by extensive ³He bubble networks and possibly precipitate particles on the grain boundaries; hopefully the bubble distribution

can be changed (for better simulation) by altering parameters of the tritium trick. The results of the investigation also show that the embrittlement resistance of vanadium alloys can be improved by adjustment of their composition and/or microstructure.

5.4	Tensile Properties of Helium-Injected V-15Cr-5Ti After Irradiation in EBR-II (Oak Ridge National Laboratory)	99
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Miniature specimens of V-15Cr-5Ti were prepared in the annealed condition and with 10, 20, and 30% cold work. The annealed specimens were cyclotron injected with helium and irradiated in sodium in EBR-II. The cold-worked specimens were irradiated in EBR-II but not helium injected. The specimens were irradiated at 400, 525, 625, and 700°C and received a fluence of 4.1 to 5.5 × 10²⁶ neutrons/m² (E > 0.1 MeV). Tensile testing revealed very significant embrittlement as a result of the neutron irradiation but a much smaller change, mostly at 400°C, resulting from helium injection.

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7.1	Charpy Impact Test Results of Ferritic Alloys at a Fluence of 6 × 10 ²² n/cm ² (Westinghouse Hanford Company)	106
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Charpy impact tests on specimens in the Ad-2 reconstitution experiment were completed. One hundred and ten specimens made of UT-9 base metal, 9 Cr-1 Mo base metal and 9 Cr-1 Mo weldment at various heat treatment conditions were tested in temperature range from -73°C to 280°C. The specimens were irradiated from 390°C to 550°C and the fluence of the specimens reached 6 × 10²² n/cm². This is the first time that the transition behavior of ferritic alloys at high fluence was obtained. This is also the first time that comprehensive results on the irradiated 9 Cr-1 Mo weldment are available.

The test results show a small additional shift in transition temperature for HT-9 base metal irradiated at 390°C and 450°C as the fluence was raised to 6 × 10²² n/cm². At higher irradiation temperatures, however, the shift in transition temperature is less conclusive. Further reduction in USE was observed at higher fluence for all the irradiation temperatures. There is no apparent fluence effect for 9 Cr-1 Mo base metal at all the irradiation temperatures studied.

Contrary to the previous finding on HT-9 base metal and weldment, the 9 Cr-1 Mo weldment shows a higher transition temperature (+60°C) and a higher USE (+100%) as compared to the 9 Cr-1 Mo base metal for the same irradiation conditions.

Significant improvement of both DBTT and USE on HT-9 base metal specimens fabricated from normalized and tempered plate stock was observed over the HT-9 base metal specimens fabricated from mill-annealed bar stock.

Overall, the highest DBTT encountered for UT-9 alloys is 141°C for specimens irradiated at 390°C. The effect on transition temperature due to additional neutron exposure appear; to be saturated at 6 × 10²² n/cm². The highest DBTT encountered for 9 Cr-1 Mo alloys, on the other hand, is 91°C for the weldment and 45°C for base metal both irradiated at 390°C.

7.2	Effect of Nickel Content on the Aging and Irradiation Response of Impact Properties of 9 Cr-1 MoVNb and 12 Cr-1 MoW in the Absence of Internal Helium Effects (Oak Ridge National Laboratory)	120
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Impact testing was completed on aged and EBR-II irradiated 9 Cr-1 MoVNb and 12 Cr-1 MoW steels, each with and without small additions of nickel. Only limited property changes resulted from aging or irradiating to 12 dpa in the temperature range of 450°C to 550°C. Irradiation of the 12 Cr-1 MoW at 390°C with and without nickel produced severe degradation of impact properties. Nickel additions affected the unirradiated material properties, but subsequent radiation-induced changes were similar regardless of nickel content.

7.3	Effects of Irradiation on Low Activation Ferritic Alloys (Hanford Engineering Development Laboratory)	128
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A series of low activation ferritic alloys has been designed, fabricated, irradiated in MOTA 1B, and tested and examined following irradiation. The series consists of alloys similar to 2 1/4 Cr-1 Mo with vanadium substituted for molybdenum, alloys similar to 9 Cr-1 Mo with tungsten and/or vanadium substituted for molybdenum and alloys similar to HT-9 with tungsten and/or vanadium substituted for molybdenum. The results demonstrate that low activation alloys can be successfully produced in the ferritic alloy class. The 2 1/4 Cr-V alloys develop excessive irradiation hardening due to precipitation following irradiation at 420°C and the 2 1/4 Cr-V and 9 Cr-V/W alloys developed excessive softening due to precipitate coarsening and dislocation recovery following irradiation at 585°C. In comparison, the 12 Cr-W-V

alloy appears to have excellent properties; α' precipitation at 420°C in-reactor did not significantly increase strength and reasonable strength was maintained after irradiation at 585°C probably in part due to intermetallic precipitate development.

7.4	The Development of Ferritic Steels for Fast Induced-Radioactivity Decay (Oak Ridge National Laboratory)	141
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Tempering studies were conducted on eight heats of normalized chromium-tungsten steel that contained variations in the composition of chromium, tungsten, vanadium, and tantalum. Hardness measurements and optical metallographic observations were used to determine alloying effects on tempering resistance between 650 to 780°C. The results were compared to results for analogous chromium-molybdenum steels.

7.5	Rapid Solidification of Candidate Ferritic Steels (Massachusetts Institute of Technology and Industrial Materials Technology)	147
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HT-9 and 9 Cr-1 Mo steels were rapidly solidified by the liquid dynamic compaction (LDC) process and 2 1/4 Cr-1 Mo steel was prepared by the ultrasonic gas atomization (USGA) process. The consolidation was performed in the ferritic temperature range in order to minimize segregation. The alloys will be tested at ORNL using 1/3 CVN test specimens and the results will be compared with those for conventionally processed alloys.

7.6	Poisson's Ratio Measurements of Martensitic Stainless Steels (Westinghouse Hanford Company)	152
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Poisson's ratio measurements using an ultrasonic technique over the temperature range room temperature to 600°C are reported for HT-9. As much as a 0.1 variation is found as a result of specimen orientation. However, based on comparisons from the literature, it is concluded that such variations are common and are not due to orientation effects. Further measurements will be needed before a design equation for the MHFES can be generated.

7.7	Effects of Irradiation on the Fracture Toughness of HT-9 (Westinghouse Hanford Company) . . .	15h
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Compact tension specimens of HT-9 irradiated at 390, 450 and 500°C were tested at 90, 205 and 450°C. Test results showed that both test and irradiation temperatures have insignificant effects on the fracture toughness of HT-9. However, the tearing modulus increases substantially with increasing irradiation temperature. In addition, the toughness of HT-9 at 205°C where a toughness trough was observed for unirradiated HT-9 remained unchanged after irradiation to a fluence of 5.5×10^{22} n/cm².

7.8	Microstructural Examination of Several Commercial Alloys Neutron Irradiated to 100 DPA (Westinghouse Hanford Company)	161
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Microstructural examination of ferritic and austenitic commercial alloys neutron irradiated to ~100 dpa confirms that ferritic alloys are very low swelling but precipitate development can be very complex. Austenitic alloys can be very high swelling but no clearly defined microstructural differences could be found between two alloys of similar composition but very different swelling response or between alloys of very different composition but with similar swelling. Differences are ascribed to differences in the onset of swelling.

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8.1	Response of Selected High Strength High Conductivity Copper Alloys to Simulated Fusion Irradiation and Temperature Conditions (Westinghouse R&D Center and University of Pittsburgh and McDonnell Douglas Company)	171
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Microstructural changes are reported for a solid solution cold work strengthened alloy (AMZIRC), a precipitation strengthened alloy (Beryllium Copper) and a dispersion hardened alloy (Al-60) after dual ion irradiation at fluences to ~5 dpa at 450°C and 500°C. The AMZIRC and Al-60 alloys are from the same heats being examined by LANL in a EBR-11 neutron irradiation program. Void swelling at a level of ~1-5% per dpa is observed in selected areas of the AMZIRC alloy that have experienced texture dependent recovery by dislocation annihilation and rearrangement. Coarsening of the G.P. zones and accelerated precipitate growth are observed in the INESCO supplied beryllium copper alloy. The mechanically alloyed Al-60 is stable and the misfit strains at the interfaces between the Al₂O₃ particles and the Cu matrix are retained. These results are used to extend a proposed test matrix and to suggest (preliminary) alloy modifications for future neutron irradiation experiments.

8.2	Effects of Neutron Irradiation at 450°C and 16 dpa on the Properties of Various Commercial Copper Alloys (Hanford Engineering Development Laboratory)	183
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High-purity copper and eight copper alloys were irradiated to -16 dpa at ~450°C in the MOTA experiment in FFTF. These alloys were also examined after aging at 400°C for 1000 hours. The radiation-induced changes in the electrical conductivity, tensile properties and density were measured and compared to those of the aged materials. The changes in conductivity can be either positive or negative depending on the alloy. Changes in tensile properties of most, but not all, of the alloys seem to be primarily dependent on thermal effects rather than the effect of atomic displacements. Radiation at 450°C induced changes in density varying from 0.66% densification to 16.6% swelling. The latter occurred in Cu-0.1% Ag and implies a swelling rate of at least 1%/dpa.

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9.1	Corrosion of Low Activation Austenitic Alloys and Standard Fe-12 Cr-1 MoVW Steel in Thermally Convective Lithium (Oak Ridge National Laboratory)	189
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Manganese-containing steels (15-30 wt %) experienced substantial corrosion when exposed to thermally convective lithium at 500°C with the 30 wt % Mn steels showing unacceptably high corrosion losses. The mass transfer of 12 Cr-1 MoVW steel at 600°C in lithium was significantly less than that of type 316 stainless steel exposed under similar conditions.

9.2	Corrosion of Type 316 Stainless Steel and 12 Cr-1 MoVW Steel in Flowing Pb-17 at. % Li (Oak Ridge National Laboratory)	196
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Type 316 stainless steel and 12 Cr-1 MoVW steel were exposed to Pb-17 at. % Li for about 2000 h at 500°C. Type 316 stainless steel was severely corroded with deep penetration of the alloy by lead-lithium. The 12 Cr-1 MoVW steel did not suffer penetration and corroded uniformly.

9.3	Environmental Effects on Properties of Structural Alloys in Flowing Lithium (Argonne National Laboratory)	200
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Data on the surface composition of type 316 stainless steel exposed to lithium under various conditions of time, temperature, and nitrogen content in lithium are presented. The results indicate that the depletion of chromium from the steel depends on the exposure conditions. The depletion of nickel is rapid and independent of temperature and lithium purity.

9.4	Compatibility of Li ₂ O in a Flowing Helium Environment (Argonne National Laboratory)	205
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Sintered Li₂O pellets exposed to flowing helium containing 2 ppm each of H₂O and H₂ show weight losses at 550 and 650°C and a weight gain at 450°C. The rates of weight loss are -3 orders of magnitude greater than those predicted from equilibrium reaction kinetics.

9.5	Environmental Effects on the Properties of Vanadium-Base Alloys (Argonne National Laboratory)	209
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Exposures of V-15Cr-5Ti, V-20Ti, and VANSTAR-7 specimens for 2000 h to pressurized flowing water containing 4 ppm dissolved O₂ at 288°C have been completed. Both the V-20Ti and VANSTAR-7 alloys formed nonadherent and nonprotective corrosion products, and both alloys exhibited relatively high corrosion rates. The V-15Cr-5Ti alloy formed a thin adherent film, and its corrosion rate was more than two orders of magnitude lower. Further results obtained in a scanning Auger microprobe study of sulfur segregation in vanadium-base alloys indicate that the extent of intergranular segregation varies markedly with different heats of material.

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According to a thermodynamic model of intergranular stress corrosion cracking, PCA is predicted to have about the same cracking susceptibility as type 316 stainless steel. However, microstructural manipulation of the EA alloy may allow improvement in the resistance to sensitization and, therefore, to intergranular stress corrosion cracking.

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