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# Alloy Development for Irradiation Performance

Quarterly Progress Report  
For Period Ending March 31, 1981

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**U.S. Department of Energy**  
Office of Fusion Energy

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	<p><i>The first publication package of data sheets for the MHFES has been released and contains revisions in the handbook format along with procedures for incorporating data sheets in the handbook and data sheets describing the effect of irradiation on the fatigue strength of 20% cold worked stainless steel. A second publication package contains data sheets on fatigue crack growth of 20% cold worked stainless steel, electrical resistivity of stainless steel, and tritium permeability of stainless steel and will be released next month. Data sheets on the properties of G-10CR glass laminate, ferritic steels, solid tritium breeding compounds, carbon and graphite, liquid lithium, and irradiation induced swelling and creep of 20% cold worked stainless steel are in work.</i></p>	
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	<p><i>A technical memorandum covering this work has been completed and printed: Neutronic Calculations for the Conceptual Design of an In-Reactor Solid Breeder Experiment, TRIO-01, ORNL/TM-7758 (March 1981).</i></p>	
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fluence of  $4.05 \times 10^{25}$  neutrons/m<sup>2</sup>, a total fluence of  $1.21 \times 10^{26}$  neutrons/m<sup>2</sup>, and a calculated response in type 316 stainless steel of 3.07 dpa and 21.01 at. ppm He (not including 2.0 at. ppm He from <sup>10</sup>B). Using these data and previous calculations, real time projections have been made to estimate the dates that core pieces should be changed and the first samples removed.

The calculations required to validate the reduction of gamma heating rates within the ORR-MFE-4A experimental capsule due to the use of thermal flux reducing core pieces have been completed. These calculations predict substantial decreases in the heating rates that are unavoidable since they are caused by the thermal flux reductions that are required to maintain the desired ratio of helium production to displacement damage over the lifetime of the experiment.

**2.4 Operation of the ORR Spectral Tailoring Experiment** . . . . . 18  
**ORR-MFE-4A (Oak Ridge National Laboratory)** . . . . .

The ORR-MFE-4A experiment consists of two test regions designed to irradiate type 316 stainless steel and Path A FCA at temperatures of 330 and 400°C. The experiment was installed in the Oak Ridge Research Reactor on June 10, 1980, and as of March 31, 1981, has operated for an equivalent 195 d at 30 MW reactor power, with maximum specimen temperatures in each region of 330 and 400°C, respectively.

The failure of test region thermocouples required the removal of the capsule from the reactor, but the successful removal of the broken flux monitor tube and its replacement with a multiple junction central thermocouple permitted reinstallation of the capsule two months after removal. Satisfactory operation is once more under way.

**3. PATH A ALLOY DEVELOPMENT – AUSTENITIC STAINLESS STEELS** . . . . . 23

**3.1 Fatigue of HFIR-Irradiated 20%-Cold-Worked Type 316**  
**Stainless Steel at 550°C (Oak Ridge National**  
**Laboratory)** . . . . . 24

Specimens of 20%-cold-worked type 316 stainless steel were irradiated at 550°C in the High Flux Isotope Reactor. Low-cycle, 550°C vacuum fatigue tests were performed on specimens with a damage level of approximately 9 dpa and containing approximately 400 at. ppm He. The preliminary results show essentially no effect of irradiation at these temperatures. Furthermore, the fatigue life of the irradiated material tested at 550°C was very similar to the unirradiated life of the same heat of material tested at 430°C.

- 3.2 Microstructural Development in 20%-Cold-Worked Type 316 Stainless Steel and Titanium-Modified Type 316 Stainless Steel Irradiated in the HFIR: Fluence Dependence of the Cavity Component (Oak Ridge National Laboratory) . . . . . 28

*Irradiation of 20%-cold-worked type 316 stainless steel and titanium-modified type 316 stainless steel in HFIR results in considerable microstructural development at 285°C and above at fluences as low as 7.7 dpa (380 at. ppm He). The microstructural development is significantly different in the two alloys; however, the cavity behavior is intimately related to the complex precipitation and dislocation behavior in both alloys. Total cavity swelling in CW 316 at 375 and 475°C is first observed to increase and then decrease before increasing monotonically with increasing fluence. Swelling increases steadily with fluence for CW 316 at 565°C and above. The steady-state swelling rate of CW 316 appears nearly temperature independent at a value of about 0.1%/dpa, with a minimum swelling rate of about 0.07%/dpa at 475°C. By comparison, steady-state swelling rates for the same material irradiated in the Experimental Breeder Reactor-II are quite sensitive to temperature and have a maximum rate of about 0.5%/dpa at 500-550°C. At fluences up to 16 dpa, the swelling of CW 316 + Ti appears to increase with increasing fluence at rates that vary from about 0.008%/dpa at 375°C to about 0.002%/dpa at 565°C, 10 to 40 times lower than CW 316 irradiated at the same conditions. Cavities are the dominant grain-boundary feature for both alloys at temperatures above 550°C and fluences of about 8 dpa or greater. The grain-boundary cavities are about a factor of 2 smaller in CW 316 + Ti than in CW 316. In CW 316 irradiated to higher fluences at temperatures above 600°C, the grain-boundary cavities contribute significantly to the total swelling.*

- 3.3 Microstructural Development in 20%-Cold-Worked Types 316 and 316 + Ti Stainless Steels Irradiated in HFIR: Temperature and Fluence Dependence of the Dislocation Component (Oak Ridge National Laboratory) . . . . . 57

*To be reported in the next quarterly report.*

- 3.4 Tensile Properties and Swelling of 20%-Cold-Worked Type 316 Stainless Steel Irradiated in HFIR (Oak Ridge National Laboratory) . . . . . 58

*Immersion density and elevated-temperature tensile properties were determined on 20%-cold-worked type 316 stainless steel irradiated at approximately 285, 370, 470, 560, and 620°C. Irradiation was to fluences up to  $3.9 \times 10^{26}$  neutrons/m<sup>2</sup> (>0.1 MeV); this fluence resulted in displacement damage levels up to 29 dpa and helium concentrations up to 1900 at. ppm. Tensile tests were at temperatures near the irradiation temperatures (300, 350, 450, 575, and 600°C).*

*Immersion density results indicated that swelling increased with increasing irradiation temperature. A maximum swelling of 1.2% was observed after the 620°C irradiation. Irradiation at the lowest temperature (284°C) increased the strength. At 370°C the strength went through a maximum with increasing neutron fluence. At the higher irradiation temperatures (470, 575, and 620°C) the strength decreased with increasing fluence. Ductility (both total and uniform elongation) generally reflected the strength behavior: an increase in strength resulted in a decrease in ductility. The large decrease in ductility at 575°C that was noted in a previous experiment was not found in the present work.*

3.5 Microstructural Development and the Effects of Helium in Type 316 Stainless Steel Irradiated in HFIR and in EBR-II (Oak Ridge National Laboratory) . . . . . 70

*The effects of different continuous helium generation rates on microstructural evolution of type 316 stainless steel is examined by comparing samples of a single heat, irradiated in HFIR and in EBR-II at similar temperatures and displacement damage levels. The effect of different initial helium concentrations is examined by comparing samples with 0 and 110 at. ppm preinjected helium irradiated in EBR-II. Both comparisons show important effects of helium on the microstructure developed during irradiation. In solution-annealed type 316 stainless steel, increased helium favors fine bubble formation, instead of coarse void formation and increased precipitation. In the 20%-cold-worked type 316 stainless steel the helium results in similar effects and also considerable dislocation recovery. High-magnification examination of samples irradiated in EBR-II shows that helium bubbles at dislocations or precipitate interfaces precede void formation at these same sites. Helium preinjection amplifies the bubble nucleation effect during EBR-II irradiation, reduces void swelling, and increases precipitation, analogous to the effect observed during continuous, high-rate helium generation. The increased helium generation rate also causes more grain-boundary cavity formation.*

4. PATH B ALLOY DEVELOPMENT — HIGHER STRENGTH Fe–Ni–Cr ALLOYS . . . . . 93  
*No contributions.*

5. PATH C ALLOY DEVELOPMENT— REACTIVE AND REFRACTORY ALLOYS . . . 95

5.1 Mechanical Property Evaluations of Path C Vanadium Scoping Alloys (Westinghouse Electric Corporation) . . . . . 96

*Tensile testing of sheet specimens of the three Path C vanadium Scoping Alloys has been completed at room temperature, 450, 500, 550, 600, 650, 700, and 750°C. The results of these tensile tests are in good agreement with values reported previously in the literature for other heats of these alloys. A series of creep/stress-rupture tests has been initiated. To date, a single specimen of each alloy is undergoing testing at 650°C in ultrahigh vacuum (pressure < 10<sup>-8</sup> torr). Stresses were selected to produce rupture in approximately 1000 hours; these stresses are 148, 276, and 414 MPa for the alloys V-20Ti, VANSTAR-7, and V-15Cr-5Ti, respectively. At the time of this report total test times of 680, 1170, and 550 hours, respectively, have been accumulated for these initial tests.*

5.2 Corrosion of Titanium Alloy Specimens from AD-1 Experiment (Hanford Engineering Development Laboratory) . . . . . 106

*The three capsules comprising the AD-1 experiment were designed for irradiation temperatures of 394°C, 450°C and 550°C and have been irradiated in EBR-11 until fluences greater than  $4 \times 10^{22}$  n/cm<sup>2</sup> (E > 0.11 MeV) were attained. Corrosion was observed on several titanium alloy specimens contained in the subcapsule designed for an irradiation temperature of 550°C, and evidence has been obtained for indicating this corrosion is due to the NaK and water reaction which occurred during the cleaning of the specimens. The other two capsules have been opened using a new cleaning technique and no evidence for corrosion has been observed.*

5.3 The Effect of Hydrogen on Flaw Growth of Titanium Alloy Ti-6242s (McDonnell Douglas Corporation) . . . . . 110

*Fatigue crack growth rate tests are being conducted at room and elevated temperatures with environment hydrogen pressures from 0 to 400 Pa on Ti-6242s samples containing 50 and 530 wppm internal hydrogen. Based on these tests the following conclusions have been made: External environment hydrogen at pressures less than 400 Pa has no effect on the fatigue crack growth rate in Ti-6242s with 50 or 530 wppm H; internal hydrogen at a concentration of 530 wppm increases the crack growth rate at intermediate and high stress intensity factor levels; the crack growth rate in Ti-6242s with 530 wppm H progressively diminishes as the temperature increases from room temperature; and the crack growth rate in hydrogen charged Ti-6242s increases with decreasing cyclic load frequency.*

6. PATH D ALLOY DEVELOPMENT — INNOVATIVE MATERIAL CONCEPTS . . . . 115

6.1 The Effect of Neutron Irradiation on the Tensile Properties of Long-Range-Ordered Alloys (Oak Ridge National Laboratory) . . . . . 116

*Postirradiation tensile tests were conducted on specimens of two different long-range-ordered alloys that had been irradiated in the ORR at temperatures of 250, 350, and 550°C, to a fluence producing 3.8 dpa and 19 to 29 at. ppm He. The irradiation increased the yield strength or "hardened" the material while the ultimate strength was decreased at all temperatures except 350°C. The ductility also decreased at all test temperatures, as evidenced by the reduction in uniform elongation and the appearance of areas of intergranular fracture in scanning electron microscopy fractographs. The reason for the relatively high ductility of the specimens that were irradiated and tested at 350°C compared to those at either 250 or 550°C is not clear.*

6.2 Mechanical Properties of Iron-Base Long-Range-Ordered Alloys (Oak Ridge National Laboratory and Rensselaer Polytechnic Institute) . . . . . 125

*Creep behavior and fatigue properties of several iron-base LRO alloys were characterized as functions of stress, temperature, and alloy composition. The LRO alloys showed a very rapid change in creep rate near their critical ordering temperature,  $T_c$ . Formation of long-range order lowers the steady-state creep rate by more than 3 orders of magnitude. The alloys exhibited a rupture ductility of 3.7 to 5.6% at temperatures below  $T_c$ . Preliminary examination of fracture surfaces revealed that the low ductility is associated with nucleation, growth, and coalescence of cavities along grain boundaries. Limited creep data indicate that preparation of LRO-37 (Fe-22% V-40% Ni-0.4% Ti) from commercial-grade ferrovanadium does not degrade the creep properties of the alloy, compared to material produced from high-purity melt stock. High-frequency fatigue tests of alloy LRO-37 showed a small decrease in fatigue resistance with increasing temperature. Comparison of fatigue data among commercial alloys has demonstrated that LRO-37 is superior to type 316 stainless steel, Inconel 617, and Hastelloy X near 600°C, and superior to Inconel 617 near 400°C and at 25°C. Fractographic examination of fatigue failure surfaces in alloy LRO-37 revealed a very faceted appearance, which is partially due to cracking along annealing twin boundaries.*

6.3 Scale-up of an Iron-Base Long-Range-Ordered Alloy (Oak Ridge National Laboratory) . . . . . 135

*A contract has been negotiated for the semiproduction scale-up of an iron-base long-range-ordered alloy by a commercial source. Three ingots, each weighing approximately 18 kg (40 lb), will be supplied with nominal composition: Fe-39.5 Ni-22.4 V-0.4 Ti (wt %). Three thicknesses of sheet, and ingot material for later processing, will be produced.*

7. PATH E ALLOY DEVELOPMENT— FERRITIC STEELS . . . . . 137

7.1 The Effect of Austenitizing Time and Temperature on the Microstructure of a 12 Cr-1 Mo-0.3 V Steel (HT-9) (General Atomic Company) . . . . . 138

*To be reported in the next quarterly report.*

7.2 Tensile Properties of Ferritic Steels after Low-Temperature HFIR Irradiation (Oak Ridge National Laboratory) . . . . . 139

*Tensile specimens from small heats of ferritic (martensitic) steels based on 12 Cr-1 MoVW, 9 Cr-1 MoVNb, and the low-alloy ferritic 2 1/4 Cr-1 Mo steel have been irradiated at coolant temperature in HFIR to displacement-damage levels of up to 9.3 dpa and helium contents of 10 to 82 at. ppm. The base compositions and similar alloys to which nickel had been added for helium production are included in the irradiations.*

*During the present reporting period, irradiated specimens from a heat of 9 Cr-1 MoVNb and two heats of 9 Cr-1 MoVNb with 2% Ni were tensile tested at room temperature and 300°C. Yield strength and ultimate tensile strength of the irradiated samples displayed considerable hardening over the unirradiated condition. The increased strength was accompanied by a decreased ductility. Indications are that the hardening resulted only from the displacement damage and was not affected by the transmutation helium formed during irradiation. These results are similar to those for the 12 Cr-1 Mo-base alloys, which were previously reported.*

7.3 Preparation of Alloy HT-9 and Modified Alloy 9Cr-1Mo Reference Plates for Unirradiated and Irradiated Condition Fracture Resistance Studies (Naval Research Laboratory) . . . . . 148

*Alloy HT-9 and modified Alloy 9Cr-1Mo are being evaluated for potential applications as first wall materials in magnetic fusion reactors. One objective of the current investigations is the assessment of material notch ductility and static fracture toughness in the preirradiation and postirradiation conditions.*

Two sections of 1.7 cm thick plate from the HT-9 reference melt were heat treated by normalizing at 1050°C for 0.5 hours and tempering at 780°C for 2.5 hours. Good agreement of tensile test values with prior results for other plate sections from the melt was observed. Yield strength, tensile strength and Charpy-V (C<sub>V</sub>) upper shelf energy levels, however, are lower than those for material (rod) from the Alloy HT-9 reference melt of the Cladding/Duct Alloy Development Program.

Tensile and C<sub>V</sub> test results for a 1.3 cm thick plate from the modified Alloy 9Cr-1Mo melt are also reported.

7.4 Microstructural Examination of a Series of Commercial Ferritic Alloys Irradiated to Moderate Fluence (Hanford Engineering Development Laboratory) . . . . . 157

A series of five commercial ferritic alloys 2 1/4 Cr-1 Mo, H-11, EM-12, 416 and 430F have been examined by transmission electron microscopy following irradiation at 425°C to 5.05 x 10<sup>22</sup> n/cm<sup>2</sup> (E > 0.1 MeV) in order to provide estimates for precipitation kinetics in this class of alloys based a comparison with earlier work a similar specimens irradiated to higher fluence. Results demonstrate that Mo<sub>2</sub>C in 2 1/4 Cr-1 Mo and H-11 and an as yet unidentified phase in 416 develop very rapidly. Chi phase in EM-12 and α' phase in 430F develop more sluggishly. Therefore postirradiation mechanical property changes may be expected to saturate in 2 1/4 Cr-1 Mo, H-11, and 416 by 5 x 10<sup>22</sup> n/cm<sup>2</sup> at 425°C but changes can be expected to continue beyond 5 x 10<sup>22</sup> n/cm<sup>2</sup> in EM-12 and 430F.

7.5 Microstructural Examination of Postirradiation Deformation in 2 1/4 Cr-1 Mo (Hanford Engineering Development Laboratory) . . . . . 165

Microstructural examinations using transmission electron microscopy have been performed a a tensile specimen of 2 1/4 Cr-1 Mo in the thermal annealed condition irradiated to 6.1 x 10<sup>22</sup> n/cm<sup>2</sup> (E > 0.1 MeV) at 400°C following postirradiation deformation. It is found that large increases in yield strength and ultimate tensile strength are a result of extensive precipitation of Mo<sub>2</sub>C in weak ferrite grains and that effects of precipitation saturate by 10<sup>22</sup> n/cm<sup>2</sup> or 5 dpa.

7.6 Evidence of Segregation to Martensite Lath Boundaries in a Temper-Embrittled 12 Cr-1 Mo-0.3 V Steel (HT-9) . . . . . 174

To be reported in the next quarterly report.

**7.7 Environmental Effects on Properties of Ferritic Steels (Argonne National Laboratory) . . . . . 175**

*Several continuous-cycle fatigue tests have been conducted on Type 304 stainless steel at 755 K in lithium containing -700 wppm nitrogen. The test specimens show secondary cmcks along the entire gauge length. Similar behavior was observed for HT-9 alloy tested in lithium containing -1400 wppm nitrogen. Secondary cmcks are genemlly not observed in ferritic and stainless steels tested in a low-oxygen sodium environment. These results indicate that the concentration of nitrogen in lithium has a strong effect on the fatigue behavior of ferritic as well as austenitic steels. A 3.6-Ms (1000-h) exposure of an HT-9 specimen under constant stress in lithium at 755 K has been completed. The specimen is being examined metallographically to evaluate the combined effects of constant stress and lithium environment a the corrosion behavior of HT-9 alloy. Compatibility tests were carried out to investigate the reactivity of candidate solid breeding mterials, i.e., Li<sub>2</sub>O, LiAlO<sub>2</sub>, Li<sub>2</sub>SiO<sub>3</sub>, Li<sub>2</sub>ZrO<sub>3</sub>, and Li<sub>2</sub>TiO<sub>3</sub>, with HT-9 alloy and Type 316 stainless steel. Metallographic evaluation of the specimens is in progress.*

**7.8 The Effect of Internal Hydrogen on the Mechanical Properties of HT-9: Room Temperature (Sandia National Laboratories, Livermore, CA) . . . . . 180**

*Tensile testing of quench and tempered HT-9 in 0.10 MPa (15 psi) external hydrogen at room temperature has previously been shown to cause a reduction in ductility and change in fracture mode compared to tests in air. This report summarizes preliminary results a the effect of internal hydrogen, introduced by cathodic charging, on the tensile properties of both as-quenched and quench and tempered HT-9. Tensile specimens were cathodically charged at (0.003 A/cm<sup>2</sup>) and (0.006 A/cm<sup>2</sup>) for up to 150 minutes, immediately copper plated, and tested at mom tempemture. There was no appreciable effect of internal hydrogen on the tensile properties of quench and tempered HT-9. The hydrogen levels were believed to be greater than 30 ppm compared to 1-5 ppm in the previous gas phase testing. For the higher strength, quenched microstructure, the same charging conditions resulted in fmcture mode from cup-cone centerline cracking and void coalescence to more brittle surface cmck initiation. These results support the earlier gas phase test results that hydrogen embrittlement of quench and tempered HT-9 is not a serious concern. However, at strength levels above 700 MPa (produced here by eliminating the temper treatment) large hydrogen effects are manifest. The data reconfirm the need for hydrogen testing of irmdiation hardened samples.*

7.9 Tempering Behavior of Laser Welds in HT9 (Sandia National Laboratories, Livermore, CA) . . . . . 191

*The effect of postweld heat treatment on both the microstructure and properties of laser welds in HT9 was evaluated. High depth-to-width ratio laser welds made at a power level of 6 kW and a travel speed of 2.96 mm/sec (70 in/min) were heat treated for 1 and 2 hours at 400, 600, and 800°C (750, 1100, and 1470°F). Heat treated weldments tempered at 400°C exhibited little variation in either microhardness or microstructure relative to the as-welded properties. Tempering at 600°C markedly reduced the hardness in both the fusion zone and HAZ. The decrease in hardness was associated with the initial stages of martensite tempering and the simultaneous precipitation of alloy carbides. Heating to 800°C reduced the hardness in all regions of the weldment to the level of the base metal. Comparison of the tempering response of the laser welds with the previously reported behavior of gas tungsten-arc welds indicated that the postweld heat treatment necessary to restore adequate mechanical properties to the weld region is relatively insensitive to the welding process which is employed.*

7.10 An Auger Spectroscopic Analysis of an HT-9 Superheater Tube In-Service at 600°C for 80,000 Hours (General Atomic Company) . . . . . 208

*To be reported in the next quarterly report.*

8. STATUS OF IRRADIATION EXPERIMENTS AND MATERIALS INVENTORY . . . 209

8.1 Irradiation Experiment Status and Schedule (Oak Ridge National Laboratory) . . . . . 210

*Principal features of many ADIP irradiation experiments are tabulated. Bar charts show the schedule for recent, current, and planned experiments. Experiments are presently under way in the Oak Ridge Research Reactor and the High Flux Isotope Reactor, which are mixed spectrum reactors, and in the Experimental Breeder Reactor, which is a fast reactor.*

8.2 EIM Research Materials Inventory (Oak Ridge National Laboratory and McDonnell Douglas) . . . . . 217

*The Office of Fusion Energy has assigned program responsibility to ORNL for the establishment and operation of a central inventory of research materials to be used in the Fusion Reactor Materials research and development programs. The objective is to provide a common supply of material for the Fusion Reactor Materials Program. This*

*will minimize unintended materials variables and provide for economy in procurement and for centralized recordkeeping. Initially this inventory will focus on materials related to first-wall and structural applications and related research, but various special purpose materials may be added in the future.*

9. MATERIALS COMPATIBILITY AND HYDROGEN PERMEATION STUDIES . . . . . 221

9.1 Compatibility Studies of Ferritic Steels Exposed to Static Lithium and Type 316 Stainless Steel Exposed to Static Pb-17 at. % Li (Oak Ridge National Laboratory) . . . . . 222

*The thermodynamic tendency for carbon transfer between ferritic steels and lithium is described. The treatment predicts a much greater driving force for decarburization of 2 1/4 Cr-1 Mo steel than for Sandvik HT9 exposed to lithium, which is consistent with our experimental findings. However, the amount of carbon loss from the 2 1/4 Cr-1 Mo steel exposed to lithium would probably not be severe in the temperature range of 2 1/4 Cr-1 Mo steel application. Furthermore, decarburization by lithium can be minimized by use of a stabilized 2 1/4 Cr-1 Mo steel. Preliminary results from exposures of type 316 stainless steel to Pb-17 at. % Li at 300, 400, and 500°C indicated a significant corrosion rate at 500°C but no detrimental effects at the tensile properties of the steel.*

9.2 Corrosion of an Iron-Base Long-Range-Ordered Alloy in Flowing Lithium (Oak Ridge National Laboratory) . . . . . 229

*Data are reported on the corrosion of the long-range-ordered alloy Fe-31.8 Ni-22.5 V-0.4 Ti (wt %) exposed to lithium in type 316 stainless steel thermal-convection loops at 600 and 570°C for up to 1500 h. Corrosion rates that include a contribution from dissimilar-metal transfer of nickel from the alloy to the stainless steel are much greater than those of type 316 stainless steel previously exposed in these loops. A loosely adherent layer was observed at the LRO coupons in one of the two loops and may indicate additional complicating effects due to the dissimilar loop material.*