File Copy
Do not remove.

DOEIER-0045/6

Alloy Development for Irradiation Performance

Quarterly Progress Report
For Period Ending March 31, 1981

U.S. Department of Energy
Office of Fusion Energy

CONTENTS

fluence of 4.05 × 10^{25} neutrons/m^2, a total fluence of 1.21 × 10^{26} neutrons/m^2, 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 ^{10}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 mtes that are unavoidable since they are mused by the thermal flux reductions that are required to rmintain the desired ratio of helium production to displacement darmge over the lifetime of the experiment.

The ORR-MFE-4A experiment consists of two test regions designed to irradiate type 316 stainless steel and Path A PCA 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.

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 darmge level of approximately 9 dpa and containing approximately 400 at. ppm He. The preliminary results show essentially no effect of irmdiation at these temperatures. Furthermore, the fatigue life of the unirradiated material tested at 550°C was very similar to the unirradiated life of the same heat of rmterial tested at 430°C.

Irradiation of 20%-cold-workedtype 316 stainless steel and titanium-modified type 316 stainless steel in HPIR 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 mtes for the same material irradiated in the Experimental Breeder Reactor-II are quite sensitive to temperature and have a maximum mte 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 ftuence at mtes 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.

To be reported in the next quarterly report.

Imersion 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 × 10^{26} neutrons/m^2 (>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).

Imersion 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 irmdiation temperatures (470, 575, and 620°C) the strength decreased with increasing fluence. Ductility (both total and uniform elongation) genemlly 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.

The effects of different continuous helium generation mtes on microstructural evolution of type 316 stainless steel is examined by comparing samples of a single heat, irmdiated in HFIR and in EBR-11 at similar tempemtures and displacement darmge levels. The effect of different initial helium concentmtions 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 irmdiation. 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-11 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-11 irmdiation, reduces void swelling, and increases precipitation, analogous to the effect observed during continuous, high-mte helium generation. The increased helium generation mte also causes more grain-boundary cavity formation.

No contributions.

Tensile testing of sheet specimens of the three Path C vanadium Scoping Alloys has been completed at mom 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 litemture 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^{-8} 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.

The three capsules comprising the AD-1 experiment were designed for irmdiation temperatures of 394°C, 450°C and 550°C and have been irradiated in EBR-11 until fluences greater than 4 × 10^{22} n/cm^2 (E > 0.11 MeV) were attained. Corrosion was observed on several titanium alloy specimens contained in the subcapsule designed for an irmdiation tempemture 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.

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 nude: External environment hydrogen at pressures less than 400 Pa has no effect on the fatigue cmck growth rate in Ti-6242s with 50 or 530 wppm H; internal hydrogen at a concentmtion of 530 wppm increases the cmck growth mte 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 mom temperature; and the cmck growth rate in hydrogen charged Ti-6242s increases with decreasing cyclic load frequency.

Postirradiation tensile tests were conducted on specimens of two different long-range-ordered attoys 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 irmdiated and tested at 350°C compared to those at either 250 or 550°C is not clear.

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 tempemture, T_c. Formation of long-range order lowers the steady-state creep mte 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 datu indicate that preparation of LRO-37 (Fe–22% V–40% Ni–0.4% Til from commercial-grade ferrovanadium does not degrade the creep properties of the alloy, compared to rmterial produced from high-purity melt stock. High-frequency fatigue tests of alloy LRO-37 showed a small decrease in fatigue resistance With increasing tempemture. 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.

A contmct has been negotiated for the semiproduction scale-up of an iron-base long-mnge-ordered alloy by a commercial source. Three ingots, each weighing approximately 18 kg (40 lb), All 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.

To be reported in the next quarterly report.

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 irmdiated at coolant temuemture in HFIR to displacement-damage levels of up to 9.3 dpa and helium contents of 10 to 82 at. ppm. The Ease 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 mom tempemture and 300°C. Yield strength and ultirmte tensile strength of the irmdiated 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 darmge and was not affected **by** the transmutation helium formed during irmdiation. These results are similar to those for the 12 Cr-1 Mom-base alloys, which were previously reported.*

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

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 tmvel 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.*

To be reported in the ***next*** *quarterly report.*

Principal features of many ADIP irmdiation 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.

The Office of Fusion Energy ***has*** *assigned progmm responsibility to ORNL for the establishment and operation of a centml inventory of research mterials to be used in the Fusion Reactor Materials research and development progmms. The objective is to provide a common supply of material for the Fusion Reactor Materials Progmm. This*