DOE/ER-0045/11

Alloy Development for Irradiation Performance

Semiannual Progress Report
For Period Ending September 30, 1983

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

CONTENTS

FOREWORD iii

1. ANALYSIS AND EVALUATION STUDIES 1

1.1 Materials Handbook for Fusion Energy Systems (McDonnell Douglas Astronautics Company and Westinghouse Hanford Company) 2

During the past six months the handbook effort has been towards developing data sheets. For this period the effort has been to establish a continuous flow of data sheets into the handbook. Substantial progress has been made towards the achievement of this goal with the publication of 7 new data pages covering the properties of HT-9 and 316 stainless steel as well as the establishment of a backlog of new data sheets in various stages of preparation.

1.2 Control of Activation Levels to Simplify Waste Management of Fusion Reactor Ferritic Steel Components (Oak Ridqe National Laboratory) 4

Decay of steel activity within tens of years could simplify mste disposal or even permit recycle. For mterial recycle, N, Al, Ni, Cu, Nb, and Mo must be excluded. For shallow land burial, initial concentration limits include (in at. ppm) Ni, <20,000; Mo, <3650; N, <3650; Cu, (2400; and Nb, <1.0. Other constituents of steels Will not be limited.

1.3 Alloy Development for Fast Induced Radioactivity Decay for Fusion Reactor Applications (Oak Ridge National Laboratory) 11

waste management could be simplified by developing steels that contain only elements that produce mdioactive isotopes that decay to low levels in a reasonable time. The development of suck steels by elemental substitutions for molybdenum in Cr–Mo steels a d nickel and molybdenum in austenitic stainless steels is discussed.

1.4 Magnetic Forces on a Ferromaqnetic HT-9 First Wall/Blanket and Coolant Pipe (GA Technologies) 21

Magnetic stresses a first wall and blanket modules and a coolant pipe were calculated for a STARFIRE design made of HT9. Coolant pipe stresses depend on distance from the magnet and can be reduced by suitable support. Stresses on the first wall and blanket are not great and can be accommodated by slight design changes.

2. TEST MATRICES, EXPERIMENT DESCRIPTIONS, AND METHODS DEVELOPMENT 29

2.1 Neutron Source Characterization for Materials Experiments (Argonne National Laboratory) 30

Damage and gas production calculations are summarized for the CTR 31, 32, 34, and 35 irradiations in HFIK. Neutron dosimetry measurements and damage calculations are also presented for the T2 and RB1 irradiations in HFIK. The helium production from nickel has been revised according to new cross section evaluations for ^{59}Ni. The status of all other experiments is summarized.

2.2 Neutronic Calculations in Support of the ORR-MFE-4A and -48 Spectral Tailorinq Experiments (Oak Ridge National Lahoratory) 38

The calculated fluences from the ongoing three-dimensional neutronics calculations are being scaled to agree with experimental data. As of September 30, 1983, this treatment yields 121.6 at. ppm He (not including 2.0 at. ppm He from ^{10}B) and 8.25 dpa for type 316 stainless steel in ORR-MFE-4A a d 69.3 at. ppm He and 5.13 dpa in ORR-MFE-4B.

2.3 Operation of the ORR Spectral Tailoring Experiments ORR-MFE-4A AND ORR-MFE-4R (Oak Ridge National Laboratory) 40

The specimens contained in the ORR-MFE-4A experiment have operated for an equivalent of 680 d at 30 MW reactor power, with temperatures of 400 a d 330°C. The ORR-MFE-4B capsule was disassembled; the test specimens were transferred to a new mpsule assembly a d reinetalled in the ORR on July 19, 1983. It has operated for an equivalent of 480 d at 30 MW reactor power with temperatures of 500 and 600°C.

2.4 Summary of EBR-II AD-2 Ferritic Irradiation Experiment (Westinghouse Hanford Company) 42

No contribution.

2.5 Disk-Bend Ductility Tests for Irradiated Materials (Oak Ridge National Laboratory) 43

We nwdified the HEDL disk-bend test machine and are using it to qualitatively screen alloys that are susceptible to embrittlement caused by irradiation. Tests designed to understand the disk-bend test in relation to a uniaxial test are discussed. Selected results of tests of neutron-irradiated material are also presented.

3. PATH A ALLOY DEVELOPMENT — AUSTENITIC STAINLESS STEELS 52

3.1 Improved Swellinq Resistance for PCA Austenitic Stainless Steel Under HFIR Irradiation Through Microstructural Control (Oak Ridge National Laboratory) 52

Swelling evaluation of PCA variants and 20%-cold-worked (N-Lot) type 326 stainless steel (CW 316) at 300—600°C was extended to 44 dpa. Swelling was negligible in all the steels at 300°C after -44 dpa. At 500 to 600°C, 25%-cold-worked PCA showed better void swelling resistance than type 316 at -44 dpa. There was less swelling variation among alloys at 400°C, but again 25%-cold-worked PCA was the best.

3.2 Microstructural Design of PCA Austenitic Stainless Steel for Improved Resistance to Helium Embrittlement Under HFIR Irradiation (Oak Ridqe National Laboratory) 60

Microstructural variants of PCA and two heats of 20%-cold-worked type 316 stainless steel were irradiated in HFIR and tested for embrittlement resistance with the disk-bend test. No embrittlement was observed for irradiation to ~44 dpa and 3000 to 3600 at. ppm He at 300 and 400°C. All materials Were brittle after similar irradiation at 600°C. The best embrittlement resistance for irradiation to 44 dpa at 500°C and to 22 dpa at 600°C was found in PCA variants that contained grain boundary MC particles produced prior to irradiation.

4. PATH B ALLOY DEVELOPMENT — HIGHER STRENGTH Fe-Ni-Cr ALLOYS 66

4.1 An Evaluation of Fatigue Properties of HFIR-Irradiated Nimonic PE-16 at 430°C (Oak Ridge National Laboratory) 67

Nimonic PE-16 was irradiated in the HFIR to 6 to 9 dpa and 560 to 1000 at. ppm He at 430°C. Postirradiation fatigue tests revealed a reduction in fatigue life by about a factor of 10 at 430°C. In contrast with AISI type 316 stainless steel, no endurance limit was observed. All irradiated specimens erhibited some intergranular fracture with an increasing tendency toward "cleavage like" intragranular fracture for low strain mnges.

5. PATH C ALLOY DEVELOPMENT — REACTIVE AND REFRACTORY ALLOYS 72

No contributions.

6. INNOVATIVE MATERIAL CONCEPTS 13

No contributions.

7. PATH E ALLOY DEVELOPMENT — FERRITIC STEELS 75

7.1 Swelling in Several Commercial Alloys Irradiated to Very Hiqh Fluence (Westinghouse Hanford Company) . . . . . .

Swelling values have been obtained from a sat of commercial alloys irradiated in EBR-II to a peak fluence of 2.5 × 10^{23} n/cm^2 (E > 0.1 MeV) or -125 dpa covering the mnge *400 to 550°C. The alloys can be ranked for swelling resistance from highest to lowest as follows: the martensitic and ferritic alloys, the niobium-based alloys, the precipitation-strengthened* iron- *and nickel-based alloys, the molybdenum alloys, and the austenitic alloys.*

7.2 Evaluation of Ferritic Alloy Fe-2 1/4 Cr-1 Mo After Neutron Irradiation — Irradiation Creep and Swelling (Westinghouse Hanford Company) 82

Iwadiation creep and swelling measurements are reported for Fe-2 1/4 Cr-1 Mo after irradiation by fast neutrons over the temperature mnge 390 to 560°C. Diameter change measurements on thin-walled pressurized tubes in a bainitic condition and density change measurements on rods in a nonstandard condition were made following irradiation in the EBR II. The iwadiation creep specimens Were irradiated to a fluence of 5.7 × 10^{22} n/cm^2 (E > 0.1 MeV) or 30 dpa and the swelling specimens were irradiated to a peak fluence of 2.4 × 10^{23} n/cm^2 or 115 dpa. These results have been used as a hsis to establish in-reactor creep and swelling correlations for 2 1/4 Cr-1 Mo in a bainitic condition. The correlations predict moderate swelling and moderate irmdiation-enhanced creep at 390°C.

7.3 Elevated-Temperature Tensile Properties of 2 1/4 Cr-1 Mo Steel Irradiated in the EBR-II, AD-2 Experiment (Oak Ridge National Laboratory) 95

The effect of irradiation on the tensile properties of 2 1/4 Cr-1 Mo steel was determined for specimens irradiated in ERR-11 at 390 to 550°C. Unirradiated control specimens and specimens aged for 5000 h at the irradiation temperatures were also tested. Irradiation to approximately 9 dpa at 390°C increased the strength and decreased the ductility compared with the unirradiated and aged specimens. Softening occurred in samples irradiated and tested at 450, 500, and 550°C.

7.4 Density Chanqe Measurements for Simple Ferritic Alloys Irradiated to Hiqh Fluences (Westinghouse Hanford Company) 103

A series of Fe-Cr-C-Mo simple alloys has been masured for density change as a function of irradiation in EBR-II over the temperature mnge 400 to 650°C to fluences as high as 2.13 × 10^{23} n/cm^2 IF > 0.1 MeV) or 105 dpa. Tke highest swelling was found in an Fe-12 Cr binary alloy, 4.72 percent, after 1.87 × 10^{23} n/cm^2 or 95 dpa at 425°C, which corresponds to a swelling mte of 0.06%/dpa. This peak swelling mte value can be used to define melting predictions for commercial ferritic alloys to 40 MWy/m^2.

7.5 Microstructure of 9 Cr-1 MoVNb and 12 Cr-1 MoVW Ferritic Steels After Irradiation at Elevated Temperatures in HFIR (Oak Ridge National Lahoratory) 108

Microstructures of 9 Cr-1 MoVNb and 12 Cr-1 MoVW were examined following irradiation in HFIR to 36 dpa at 300 to 600°C. Maximum cavity development in the form of voids occurred at 400°C with only small helium bubble formation at 500 and 600°C. Swelling was greater in 9 Cr-I MoVNb than in 12 Cr-1 MoVW. The cavity development is attributed in part to the production of helium during irradiation.

7.6 Effect of HFIR Irradiation at 55°C on Microstructure and Toughness of HT-9 (Westinghouse Hanford Company) 115

Results are reported for base metal and weld metal specimens of HT-9 and nwdified 9 Cr-1 Mo following irradiation in HFIR at 55°C to 5 dpa. The DBTT shifts in irradiated base metal specimens were 30°C for HT-9 and 90°C for 9 Cr-1 Mo with further shifts of 20°C for weld metal. Concurrently, strength as measured by hardness increased 15 percent for HT-9 and 25 percent for 9 Cr-1 Mo. The hardness increases can be attributed in part to defect clusters 1.5 to 3.0 nm in diameter at densities approaching 10^{17} cm^{-3} and also to tower mtes of mvity nucleation ahead of the propagating crack.

7.7 Fractographic Examination of HT-9 Miniature Compact Tension Specimens Tested at Low Temperatures (Westinghouse Hanford Company) 128

Fractographic examinations have been performed on a series of miniature compact tension specimens of HT-9 tested at room temperature and below. Reductions in toughness found for these specimens are shown to be associated with transgranular cleavage fracture and are a function of the plastic deformtion which occurs prior to the onset of brittle fracture.

7.8 Postirradiation Tests of ESR Alloy HT-9 and Modified 9 Cr-1 Mo Alloy from UBR Reactor Experiments (Naval Research Laboratory) 136

During this period, irradiation exposures at 300°C and 150°C to ~8 × 10^{19} n/cm^2, E > 0.1 MeV, were completed for the Alloy RT-9 plate and the modified Alloy 9 Cr-I MO plates, respectively. Postirradiation tests of Charpy-V (C_v) specimens were completed for both alloys; other specimen types included in the reactor assemblies were fatigue precracked Charpy-V (PCC_v), half-size Charpy-V, and, in the case of the mdified 9 Cr-1 Mo, 2.54-m-thick compact tension specimens.

7.9 Effect of Specimen Size and Nickel Content on the Impact Properties of 12 Cr-1 MoVW Ferritic Steel (Oak Ridge National Laboratory) 143

Charpy impact properties Were developed on sir heats of 12 Cr-1 MoVW steel with full- and subsize specimens to examine effects of nickel and chromium adjustments. These data provide a baseline for comparison of effects of irradiation on the impact properties of these alloys.

7.10 Grain Boundary Segregation and Embrittlement in a 12 Cr-1 Mo Steel Resultinq from Cooling Rate Variations (Sandia National Laboratories) 148

Segregation of embrittling elements, especially phosphorus, to the prior austenite grain boundaries in this 12 Cr-1Mo steel has been shown to reduce tensile ductility, increase grain boundary fracture, and affect hydrogen compatibility. Tests Were performed to determine the influence of variations in cooling mte during heat treatment on the tensile properties. The non-hydrogen-exposed tensile ductility increased a d the occurrence of intergranular rupture decreased as the cooling rates were increased. Phosphorus segregation at the prior austenite grain boundaries also decreased as the cooling mtes Were increased.

7.11 An Assessment of the Weldability of HT-9 Using a Y-Groove Test (GA Technologies) 152

A minimum preheat temperature of 200°C (392°F) was found necessary for gas tungsten arc (GTA) welding HT-9 plate of thickness 9.5 mm (3/8 in.) and 16 mm (5/8 in.). Thermal shock treatments produced no cmcking in welded test specimens.

7.12 Time-Temperature Characteristics of the Various Heat-Affected Zones in HT-9 Weldaments (GA Technologies) 155

Tempemtures at different distances from the fusion boundary were masured during GTA weld depositing MTS-4 filler wire on 9.52 mm (3/8 in.) thick HT-9 plate. Peak temperature measurements indicate each of the heat-affected regions to be austenitized. An exponential expression has been used to describe the cooling curves as a function of peak temperature (or distance from the fusion boundary).

7.13 Fractographic Examination of Crackinq in Multipass Welds in HT-9 (Sandia National Lahoratories) 162

No contribution.

7.14 TEM Observations of HT-9 As-Welded Weldment Microstructures (GA Technologies) 163

TEM studies of different locations in an HT-9 weldment indicated delta-ferrite (δ) occurrence, $M_{23}C_6$ precipitation at δ-martensite interfaces, fine carbide precipitation at prior austenite grain boundaries, and martensite lath and lath packet siae to be the distinguishable microstructure features observed. Furthermore, retained austenite films were observed in the weld metal and the HAZ adjacent to the weld metal that reached the highest temperature during joining. The microstructures correlate welt with the observed room temperature microhardness except for the fusion boundary in weld mtal, which exhibited a hardness drop and an unexpected minimum mount of delta-ferrite.

7.15 An Assessment of the Applicabi1ity of Critical-Stress-Critical-Distance Models of Cleavage fracture in Martensitic Steels (GA Technoloqies) 171

No contribution.

7.16 The J_{1c} Fracture Touqhness Transition Behavior of HT-9 (Westinghouse Hanford Company) 172

Small compact tension specimens of two heats of HT-9 were tested at temperatures mnging from room tempemture to −192°C. The ductile-brittle transition toughness of HT-9 was evaluated using the J-integral approach. There were two loading mtes of 2.1 × 10^{-5} m/s and 3.2 × 10^{-2} m/s. The ductile-brittle-transition temperatures of HT-9 (No. 1 heat) tested at 2.1 × 10^{-5} m/s and HT-9 (No. 2 heat) tested at 3.2 × 10^{-2} m/s Were found to be −60 and −10°C, respectively. Results showed the fracture toughness of the former was not sensitive to loading mte a d the tower-shelf toughness decreased with temperature to a J_{1c} value of 5 kJ/m^2 at −190°C. Furthermore, the values of J_{1c} were valid since the thickness of the test specimens was well above the thickness criterion.

1.17 The Fracture Toughness of Ferritic Alloys Irradiated in HFIR (Westinghouse Hanford Company) 180

The fracture toughness of HT-9 and 9 Cr-1 Mo irradiated in HFIK to 10 dpa at 50°C was evaluated using electropotential single specimen techniques. Circular compact tension specimens were tested at temperatures mnging from room temperature to 450°C. The test results were analyzed using the J-integml approach. Analyses showed that the fmcture toughness of both alloys was reduced as a result of HFIR irradiation. The degradation of irradiation toughness resistance was more severe for 9 Cr-1 Mo than HT-9. The thickness criterion for valid J_{1c} values was satisfied by the thickness of the test specimens.

8. STATUS OF IRRADIATION EXPERIMENTS AND MATERIALS INVENTORY 185

8.1 Irradiation Experiment Status and Schedule (Oak Ridge National Laboratory) 186

8.2 Fusion Program Research Materials Inventory (Oak Ridqe National Laboratory, McDonnell Douglas Company, and GA Technologies) 187

9. MATERIALS COMPATIBILITY AND HYDROGEN PERMEATION STUDIES 196

9.1 Corrosion of Path A PCA and 12 Cr-1 MoVW Steel in Thermally Convective Lithium (flak Ridqe National Lahoratory) 197

Exposure of path A PCA alloys to thermally convective lithium for 6700 h at 600 and 570°C resulted in corrosion reactions that were similar to what is observed for other austenitic alloys exposed under similar conditions. PCA corroded more rapidly than type 316 stainless steel, and the presence of nitride stringers in PCA did not affect the measured weight losses. Consideration of the weight change and surface analysis data for 12 Cr-I MoVW steel exposed to thermally convective lithium between 500 and 350°C for 10,088 h revealed that reactions with carbon and nitrogen were probably the principal corrosion processes for this alloy in this temperature mnge. Corrosion was not severe.

9.2 Corrosion of Type 316 Stainless Steel in Flowing Pb—17 at. % Li (Oak Ridge National Laboratory) 203

The corrosion of type 316 stainless steel exposed to thermally convective Pb—17 at. % Li was severe. Plugging started to occur after just several hundred hours. At 500°C, the weight loss of type 316 stainless steel exposed to P M 7 at. % Li was more than an order of magnitude greater than that of this alloy exposed to thermally convective lithium at 600°C.

9.3 Compatibility Studies of Structural Alloys with Solid Breeder Materials (Argonne National Laboratory) 205

Compatibility tests between Li_2O pellets and austenitic Type 316 stainless steel or ferritic HT-9 alloy and Fe-9 Cr-1 Mo steel in a ftowing-helium environment indicate that the reaction rates in helium containing 93 ppm H_2O are greater than in helium with I ppm H_2O. The corrosion behavior of Type 316 stainless steel is similar to that of the ferritic steels. The Li_2O pellets, exposed both with and without the alloy specimens, lose weight in flowing helium.

9.4 Corrosion and Oxidation of Vanadium-Rase Alloys in Helium Environments (Arqonne National Lahoratory) 209

The increase in weight of unalloyed V and V-5Ti, V-15Cr and V-15Cr-5Ti alloys at 725, 825 and 925 K was determined for exposure times ranging up to 1000 hours in He containing H_2 and/or H_2O impurity. The microhardness of the specimens in a transverse section was also determined after exposure for 1000 hours. These results were utilized to discuss the consequences of the selection of certain mdiation-damage resistant, V-base alloys for structural materials applications in a fusion reactor.

9.5 Environmental Effects on Properties of Structural Alloys (Argonne National Laboratory) 216

Corrosion data are presented for several austenitic and ferritic steels exposed at temperatures between 700 and 755 K in flowing lithium and Pb-I7 Li environments. Tke results indicate that dissolution mtes for both steels are an order of magnitude greater in Pb-Li than in lithium. Tensile data for cold-worked Type 316 stainless steel show that a flowing environment has no effect on the tensile properties of Type 316 stainless steel at temperatures between 473 and 773 K.