DOE/ER-0045/14

Alloy Development for Irradiation Performance

Semiannual Progress Report
For Period Ending March 31, 1985

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) 2

Substantial progress has been made towards reducing the backlog of data pages with the release of the tenth and eleventh data packages. In addition, the handbook now has the capability of total computer generation of data pages with a quality suitable for printing.

2. TEST MATRICES, EXPERIMENT DESCRIPTIONS, AND METHODS DEVELOPMENT 4

2.1 Operation of the ORR Spectral Tailoring Experiments ORK-MFE-4A AND -48 (Oak Ridge National Laboratory) 5

The specimens contained in the ORR-MFE-4A experiment have operated for a total of 1077 d at 30 MW reactor power with temperatures of 330 and 400°C. The specimens contained in the ORR-MFE-4B experiment have operated for an equivalent of 916 d at 30 MW reactor power lthrough March 24, 19851 with temperatures of 500 and 600°C.

2.2 Irradiation Experiments for The U.S./Japan Collaborative Testing Program in HFIK and ORR (Oak Ridge National Laboratory) 6

The assembly of JP-7 and -8 was completed. Capsules JP-2 through -8 are being irradiated in the HFIR. Capsule JP-I completed its irradiation for a total of 336.40 d at 100 MW reactor power.

The fabrication and assembly of ORR-MFE-6J are in progress. The assembly of MFE-7J is nearing completion. The specimens are expected to be delivered for loading by the end of March.

2.3 Preparation for Postirradiation Examination of U.S./Japan HFIR Capsules (Oak Ridge National Laboratory) 10

The first U.S./Japan HFIR capsule, JP-I, has completed its irradiation campaign and is cooling down. Preparations for PIE are under way. The new Japan Electron Optical Laboratory, Inc. (JEOL) JEM-2000FX microscope has been delivered to ORNL and is operational.

2.4 Description of the U.S./Japan Spectral Tailoring Experiment in ORR (Oak Ridge National Laboratory and Japan Atomic Energy Research Institute) 12

Two spectral tailoring capsules MFE-6J and -7J are being assembled and irradiated in ORR a8 a part of the U.S./Japan program of collaborative testing in mized-spectrum fission reactors. The test matrix includes conventional U.S. and Japanese structural alloys plus a wide variety of research alloys that lead to a basic understanding of alloy response to the fusion reactor environment.

2.5 Neutronics Calculations in Support of the ORR-MFE-4A and -48 Spectral Tailoring Experiments (Oak Ridge National Laboratory) 20

The calculated fluences from the ongoing three-dimensional neutronics calculations are being scaled to agree with experimental data. A8 of March 32, 1985, this treatment yields 201.9 at. ppm He (not including 2.0 at. ppm He from ^{10}B) and 13.19 dpa for type 316 stainless steel in ORR-MFE-4A arid 155.9 at. ppm He and 10.60 dpa in ORR-MFE-4B.

2.6 Neutron Dosimetry and Damage Calculations for the HFIR-CTR-30, -36, and -46 Experiments (Argonne National Laboratory) 22

Dosimetry measurements and damage calculations have been completed for the CTR 30, 36, and 46 experiments in HFIR. CTR 30 and 36 have the highest exposures seen to date with fluences of $1.95\times10^{23}n/cm^2$ and $1.61\times10^{23}n/cm^2$, respectively. Work is in progress on the RB2 and T3 experiments in HFIK and a prototype of the Japanese experiments in OHR.

2.7. Overview of MIT, ADIP Irradiation Experiments (Massachusetts Institute of Technology) . . . 27

Various rapidly solidified austenitic, ferritic and copper alloys have been produced at MIT for inclusion in ADIP neutron irradiation experiments. A brief summary of the alloys and their preparation and the achieved or projected irradiation parameters is provided.

2.8 Analysis of Damage Exposure Rates in the Materials Open Test Assembly of the Fast Flux Test Facility (Westinghouse Hanford Company) 31

The axial distribution of dpa/sec in stainless steel (Fe18Cr10Ni) and flux E > 0 MeV and E > 0.1 MeV Were determined from the analysis of dosimetry measurements made during MOTA Cycles 1A and 1B.

3. AUSTENITIC STAINLESS STEELS 36

3.1 Minor Compositional Variations of the Austenitic PCA to Explore MC Formation and Stability Characteristics for Improved Radiation Resistance (Oak Ridge National Laboratory and Japan Atomic Energy Research Institute) 37

The original PCA exclusively forms MC carbides in the matrix and grain boundaries during short-term aging, with a small amount of Laves appearing at longer times. The MC phase shows a surprisingly high solubility for chromium. Small laboratory melts were made which vary C, Si, Mo, and Ti alloy concentrations together with F and Nb additions. Solution-annealed (SA) alloys with (Ti t Nb), or Nb and lower Si levels, exhibited grain boundary MC formation after 166 h at 800°C, whereas other alloys formed only coarse $M_{23}C_6$. None formed intermetallics. Alloys with stable grain boundary MC formation in the SA condition showed excellent resistance to recrystallization when cold worked (CW). The MC phase composition was quite sensitive to alloying changes; combinations of Nb, C, and P alloying additions lowered the (Mo + Cr) content of the Ti-rich MC phase, particularly in small particles.

3.2 Microstructural Development in Manganese-Stabilized Austenitic Steel Irradiated in HFIR at 500 ANU 600°C TO 19 dpa (Oak Ridge National Laboratory) 49

The 12Cr/13Mn/4Ni steel (heat EP-838, made available through an exchange with the Soviet Union) was irradiated in HFIK to 19 dpa and 220 ut. ppm He. A spatially nonuniform dispersion of voids (9–19 nm in diameter) developed at 500°C, but none formed at 600°C. Many fine (2.5–6 nm in diameter) matrix bubbles were also observed at both temperatures. A dense tangle of dislocation network and Frank loops was observed at 500°C, but only loose network was found at 600°C. There was considerable precipitation at 600°C, but much less at 500°C. Coarse $M_{23}C_6$ and/or M_6C were found along subgrain boundaries at both temperatures, but many medium-coarse laths (probably Laves) were found in the matrix at 600°C, whereas only some fine particles (probably MC) were found at 500°C.

4. HIGHER STRENGTH Fe-Ni-Cr ALLOYS 52

5. REACTIVE AND REFKACTORY ALLOYS 53

5.1 The Tensile Properties of Several Vanadium Alloys After Irradiation in FFTF (Oak Ridge National Laboratory) 54

Irradiation in ^{7}Li-filled capsules at 420 and 520°C in FFTF (MOTA) with damage levels up to 13.6 dpa substantially lowered the ductility of V-15Cr-5Ti, VANSTAR-7, and V-3Ti-1Si alloys by the mechanism of radiation hardening. The VANSTAR-7 and V-3Ti-1Si alloys resisted the embrittling effects of irradiation better than the V-15Cr-5Ti alloy, regardless of the implanted helium level. Control specimens of all three alloys were not appreciably affected by thermal aging in ^{7}Li at the respective irradiation temperatures. The mechanism causing the low ductility and transgranular cleavage fracture in one V-15Cr-5Ti specimen without implanted helium requires further investigation.

5.2 Comparison of Swelling for Neutron- and Ion-Irradiated MFK Structural Materials (Argonne National Laboratory) 62

The swelling of vanadium and vanadium-base alloys, 316 stainless steel, Fe-25Ni-15Cr alloy, ferritic steels, and copper on neutron irradiation is compared with the swelling for these mterials on ion irradiation in this report. The results of this comparison are taken to show that utilization of the ion-irradiation technique provides for a discriminative assessment of the potential for swelling of candidate MFR materials.

6. INNOVATIVE MATERIAL CONCEPTS 68

7. FERRITIC STEELS 69

7.1 Microstructure of Alloy HT-9 and Modified 9Cr-1Mo Alloy Irradiated in the UBR (Naval Research Laboratory) 70

TEM disks Were prepared from tested Charpy-V specimens of Alloy HT-9 and Modified 9Cr-1Mo which had been irradiated at 149C to –0.8 × 10^{20} n/cm^2, E > 0.1 MeV. Examination of these disks showed that the irmdiation had produced small defect clusters, <10 nm in diameter, in both alloys. These defect clusters Were the apparent source of the hardening and thus the shift in ductile–brittle transition temperature previously observed for these materials. No change8 in precipitate structure could be observed for either alloy.

7.2 Microstructural/Microcompositional Analysis of Normalired-and-Tempered 9Cr-1MoVNb and 12Cr-1MoVW Steels Uoped With 2 wt % Nickel (Oak Ridge National Laboratory) 74

Normalized-and-tempered 9Cr-1MoVNb and 12Cr-1MoVW steels are tempered rmrtensite that have recovered ferrite subgrain structures with $M_{23}C_6$ and MC precipitate and a few dislocations. The 12Cr steel has more precipitate, which is almost exclusively $M_{23}C_6$; the 9Cr steel has less precipitation, but a larger fraction of fine MC. Both steels doped with 2 ut % Ni have more refined precipitate and/or subgrain structures and higher dislocation concentrations, but these changes **are** *consistent with their lower tempering temperatures relative to undoped materials. Nickel doping does not change the precipitate phases observed ($M_{23}C_6$ and MC) or the relative phase fractions, and does not appreciably affect phase compositions.*

7.3 Some Effects of Increased Helium Generation on Microstructure and Swelling of Nickel-Uoped 9Cr-1MoVNb Steel Irradiated in HFIR (Oak Ridge National Laboratory) 81

Considerable void formation was found at 400 and 500°C in 9Cr-1MoVNb-2Ni after –38 dpa (~400 at. ppm He) in HFIR, whereas some voids were found only at 400°C for similar irradiation of the same steel without nickel doping (only –30 at. ppm He). Increased helium generation tremendously increased bubble nucleation over the **mnge** *from 300 to 600°C. The irradiation also produced considerable precipitation in the 9Cr-1MoVNb-2Ni at 400 and 500°C in conjunction with void formation.*

7.4 Postirradiation Tensile Behavior of Nickel-Doped 9Cr-1MoVNb and 12Cr-1MoVW Steels (Oak Ridge National Laboratory) 87

Tensile specimens of normalized-and-tempered 9Cr-1MoVNb, 9Cr-1MoVNb-2Ni, 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels were irradiated in EBR-II to –16 dpa at 390 to 550°C. Tests were conducted at room temperature and the irradiation temperature on unirradiated, thermally aged, and irradiated specimens. The effect of irradiation depended on temperature and on the tempering treatment that the steel was given.

7.5 Elevated-Temperature Tensile Properties of 2 1/4 Cr-1Mo Steel Irradiated in the EBR-II, AD-2 Experiment (Oak Ridge National Laboratory) 99

The effect of irradiation on the tensile properties of 2 1/4 Cr-1Mo steel was determined for specimens irradiated in EBR-11 to 23 dpa at 390 to 550°C. Unirradiated control specimens and specimens aged for 10,000 h at the irradiation temperature Were also tested. Irradiation at 390°C increased the strength and decreased the ductility of the samples while softening occurred in those irradiated and tested at 450, 500, and 550°C.

7.6 Irradiation Effects in Ferritic Steels (GA Technologies) 109

Since 1980 the Alloy Development for Irradiation Performance (ADIP) task funded by the U.S. Department of Energy has been studying the 2-12Cr class of ferritic steels to establish the feasibility of using them in fusion reactor first wall/breeding blanket (FW/B) applications. The advantages of ferritic steels include superior swelling resistance, low thermal stresses compared to austenitic stainless steels, attmctive mechanical properties up to 600°C, and service histories exceeding 100,000 hours. These steels are commonly used in a mnge of microstructural conditions which include ferritic, martensitic, tempered martensitic, tainitic, etc. Throughout this paper where the term "ferritic" is used **it** *should be taken to mean any of these microstructures. The ADIP task is studying several candidate alloy systems including 12Cr-1MoWV (HT-9), modified 9Cr-1Mo, and dual. phased steels such as EM-12 and 2 1/4Cr-1Mo. These mterials are ferromagnetic (FM), body centered cubic (BCC), and contain chromium additions between 2 and 12 wt.% and molybdenum additions usually below 2%. The perceived issues associated*

with the application of this class of steel to fusion reactors are the increase in the ductile-brittle transition temperature (DBTT) with neutron damage, the compatibility of these steels with liquid metals and solid breeding mterials, and their weldability. The ferromagnetic character of these steels can also be important in reactor design. It is the purpose of this paper to review the current understanding of these BCC steels and the effects of irradiation. The major points of discussion will be irradiation induced or enhanced dimensional changes such as swelling and creep, mechanical properties such as tensile strength and various measurements of toughness, and activation by neutron interactions with structural materials.

7.7 Melting and Fabrication of Low Activation Ferritic Alloys for FFTF Irradiations (GA Technologies) 117

Two experimental heats of W-stabilized low activation ferritic steels have been melted, the chemistries analyzed, arid the heats fabricated into specimens to be included in an FFTF irradiation. A control heat of HT-Y was melted and fabricated in the Same my. Specimens include 1/3 PCVN specimens for toughness and DBTT evaluations and TEM disks. All specimens were given a heat treatment of 1050°C for 1 hr followed by an air cool, 700°C for 1 hr followed by an air cool.

7.8 Preliminary Evaluation of Microstructure and Mechanical Properties on Low Activation Ferritic Steels (GA Technologies) 120

A preliminary evaluation of the microstructure and mechanical properties of a 9Cr-2.5W-0.3V-0.15C (GA3X) low activation ferritic steel has been performed. An optimum heat treatment condition has been defined for GA3X steel. The properties and microstructure of the quenched and tempered specimens were characterized via hardness measurement and optical metallographic observation.

Quenched (1000°C/1hr) and tempered (700°C/1hr) specimens were thernully aged at 600°C up to 1,000 hrs to examine the phase stability of the alloy. X-ray diffraction analysis of the precipitates extracted from the thermally aged samples revealed nearly 100% $M_{23}C_6$ carbides la = 10.64A), with no indication of Fe_2W-type Laves phase which may cause embrittlement via segregation of detrimental impurity elements and strengthening. The impurity elements (P, S, Sb) are in small, concentration and this, combined with a fine grain size, lead us to conclude little temper embrittlement will occur.

The hot-microhardness and ductility parameter measurements were used to estimate the tensile properties at elevated temperatures. The estimated tensile strengths of GAJX steel at elevated temperatures are comparable to both 9Cr-1Mo and the modified 9Cr-1Mo steels. These preliminary results are encouraging in that they suggest that suitable low activation alloys can be successfully produced in this ferritic alloy class.

7.9 Microstructural Comparison of HT-9 Irradiated in HFIK and EBR-II (Westinghouse Hanford Company) 129

A series of specimens of HT-Y heat $1354 have been examined following irradiation in HFIH to 39 dpa at 300, 400, 500 and 600°C and following irradiation in EBR-II to 29 dpa at 390 and 500°C. HFIR irradiation was found to have promoted helium bubble formation at all temperatures and voids at 400°C. Cavitation had not been observed at lower fluence, nor was it found in EBR-II irradiated specimens. The onset of void swelling in HFIR is attributed to helium generation. The observations provide an explanation for saturation of ductile-brittle transition temperature shifts with increasing fluence.

7.10 Fractograhic Examination of Ferritic Alloy Charpy Specimens at a Fluence of 6×10^{22} n/cm^2 (Westinghouse Hanford Company) 137

The fracture surfaces of thirteen Charpy specimens have been examined by scanning electron microscopy and several of these have been measured for hardness. The specimen series irradiated in the AD-2 test, second discharge includes HT-Y base metal in two product forms, and Modified 9Cr-1Mo base metal and weld metal, fluences include 13 and 26 dpa and irradiation temperatures cover the range 390°C to 550°C. Fracture appeamnce is found to be insensitive to irradiation fluence, whereas significant differences could be found in product form. It is concluded that irradiation does not encourage a new fracture mechanism such as temper embrittlement. Failure is controlled by the microstructure generated prior to irradiation.

8. COPPER AND ITS ALLOYS 151

8.1 Swelling and Tensile Properties of Neutron Irradiated Copper Alloys for High Heat Flux Applications (Los Alamos National Laboratory) 152

Four commercial copper alloys and two elemental coppers were irradiated to EBR-11 fluences of 3 and 15 dpa at 385°C. The AMZIRC alloy and the two elemental coppers showed large swelling at the high fluence. The two precipitation hardened and cold worked alloys (AMZIRC and MZC) were softened significantly by the irradiation. The two alumina-dispersed alloys (AI-20 and Al-601 retained their mechanical strength after irradiation.

8.2 Neutron-Radiation Induced Changes in Electrical Resistivity of Copper Alloys for MFE Applications (Los Alamos National Laboratory) 161

Values of ρ_e were measured for two elemental (high-purity) coppers and four commercial copper alloys subjected in EBR-11 to neutron fluences of 0.4 arid 2.0 × 10^{26} n/m^2 at 658K. Relative to annealed controls, two alumina-dispersed alloys gave the best results: Increases in ρ_e (and 1/K) of 6-7% at the high fluence, with no significant changes (per paper 8.1 preceding) in the corresponding yield stresses.

8.3 Dual Ion Irradiation Studies of High Strength Copper Alloys (Westinghouse R&D Center and University of Pittsburgh, and McDonnell Douglas Company) 168

Microstructural changes are reported for a precipitation strengthened Cu-0.4 wt % Be-1.9 wt % Ni alloy and a dispersion hardened Cu-0.6 wt % Al (Al_2O_3) alloy after dual ion irradiation at fluences up to -20 dpa at 250°C, 300°C and 350°C. Both alloys show good swelling resistance. Peak swelling temperature for the alumina dispersion hardened alloy (AI-601 is ~350°C. For fluences of 10-20 dpa average swelling rates of 0.01% per dpa and 0.05% per dpa have been measured for irradiation without simultaneous helium injection and with a gas atom implantation Pate of K_g ~3 × 10^{-3} appm·s^{-1}. Void formation is heterogeneous and is most pronounced in grains with low no. densities of Al_2O_3 particles. The beryllium-copper alloy shows almost no swelling with the exception of a few narrow and widely spaced transgranular bands of voids at 250°C-350°C. Loss of the (100) relrod reflections in the Latter alloy is fluence and temperature dependent and reflects loss of coherency of the C.P. zones, particle restructuring and/or loss of imaging conditions due to strain field overlap from the dislocation network. Recoil resolution of the cubic γ-Al_2O_3 particles in the Al-60 is observed under certain conditions. The impact of these phase instabilities on mechanical and thermal properties is not known. It is recommended that neutron irradiation studies at 105°C-300°C be conducted to assess their importance.

9. MATERIALS COMPATIBILITY AND HYDROGEN PERMEATION STUDIES 176

9.1 Environmental Effects on the Properties of Vanadium-Base Alloys (Aryonne National Laboratory) 177

Exposures of V-15Cr-5Ti, V-20Ti, and VANSTAR-7 specimens to pressurized flowing water containing 0.03 ppm dissolved O_2 at 288°C indicate that V-15Cr-5Ti again has the best corrosion resistance. Further scanning Auger microprobe studies of sulfur segregation in vanadium-base alloys reveal no clear correlation between segregation and the variables examined.

9.2 Corrosion of Austenitic and Ferritic Steels in Flowing Lithium Environment (Argonne National Laboratory) 182

Corrosion data on weight loss and internal penetration are presented for ferritic and austenitic steels in flowing lithium at 372, 427, and 482°C. Results from metallographic examination of the lithium-exposed specimens are presented. The influence of time, temperature, and lithium purity on the dissolution behavior of the alloys is discussed.

9.3 Environmental Effects on Properties of Structural Alloys in Flowing Pb-17Li (Argonne National Laboratory) 192

Corrosion data are presented for ferritic HT-9 and Fe-9Cr-1Mo and austenitic Type 316 stainless steel in flowing Pb-17Li at 423, 427, and 454°C. Tensile properties of HT-9 alloy in a flowing Pb-17Li environment at temperatures between 270 arid 450°C are also reported.

9.4 Thermal Convection Loop Studies of the Lithium Corrosion of Low Activation Austenitic Alloys and Standard Fe-12Cr-1MoVW Steel (Oak Ridge National Laboratory) 197

Further data for Cr-Mn steels in thermally convective lithium showed that corrosion in such systems was complicated by multiple simultaneous corrosion processes. The dominant corrosion reaction during higher temperature exposure of 12Cr-1MoVW steel to thermally convective lithium ms thermal gradient mass transfer.

9.5 Corrosion of Type 316 Stainless Steel and 12Cr-1MoVW Steel in Thermally Convective Pb-17 at. % Li (Oak Ridge National Laboratory) 205

Observations of the effects of the cleaning procedure used to remove residual Pb-17 at. % Li from type 316 stainless steel revealed a considerable variability in the amount of corrosion layer that was also removed. No localized corrosion or lead-lithium penetration was noted for 12Cr-1MoVW steel exposed to thermally convective Pb-17 at. % Li for 3078 h at temperatures up to 500°C.

10. STATUS OF IRRADIATION EXPERIMENTS AND MATERIALS INVENTORY 208

10.1 Irradiation Experiment Status and Schedule (Oak Ridge National Laboratory) 209

10.2 Fusion Program Research Materials Inventory (Oak Ridge National Laboratory, McDonnell Douglas Astronautics Company, and GA Technologies) 216