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Alloy Development for Irradiation Performance

Quarterly Progress Report
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U.S. Department of Energy
Assistant Secretary for Energy Technology
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1.1 Materials Handbook for Fusion Energy Systems (McDonnell Douglas Astronautics Company and Hanford Engineering Development Laboratory) . . . . . .

The handbook activity is well underway with requests for data sheets being issued and initial information sheets relating to format, contents, and objectioe completed and ready for publication. The schedule for publication of the data sheets on Austenitic stainless steel is targeted for September 1, with the sheets ready for review on July 15. Discussions with M. L. Grossbeck, who has lead responsibility for the preparation of the austenitic stainless steel data sheets, indicates that work is currently progressing on schedule. Requests for data sheets and/or assistance in developing correlations have been directed to the DAFS and PMI tusk groups. The DAFS group has been asked to develop correlations relating dpa, He, and MW•Yr/m^2. The PMI group has been requested to develop a uniform set of sputtering yields and a model or approach to predict the particle fluxes to the first wall, limiter, and divertor collector plates.

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2.2 Progress on the ORR-MFE-4 Spectral Tailoring Experiment (Oak Ridge National Laboratory) 14

The hardware for ME-4A (300 and 400°C) is now fabricated and assembled to the point of specimen loading. Only pressurization of the Prime Candidate Alloy tubes remains to be completed. All other specimens are ready for loading. Experiment MFE-4A is scheduled to be installed in the ORR in the latter half of April 1980. Experiment MFE-4B (500 and 600°C) is scheduled for insertion in June 1980.

2.3 Neutronic Calculations for the Conceptual Design of an "In-Pile" Solid Breeder Experiment IPSB-01 (Oak Ridge National Laboratory) 16

A model has been developed that represents the solid breeder experiment and the adjacent fuel regions of the ORR core. This model and appropriate data sets will be used in transport calculations to provide tritium production and nuclear heating rates for operation of the experiment in the ORR.

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Previous work designed microstructures that might provide optimum irradiation response of the Path A PCA. The fabrication problems encountered with producing homogeneous Path A PCA material were solved, and the response to short-tern thermal aging was investigated. The specification of desired microstructures was revised to be consistent with the aging results; practical fabrication sequences and TMTs were suggested. The current work examines the success of the revised fabrication and TMT in producing the desired microstructures and characterizes the microstructures achieved.

3.2 High-Cycle Fatigue Testing of HFIR-Irradiated 20%-Cold-Worked Type 316 Stainless Steel (Oak Ridge National Laboratory) 33

Work is now in progress to extend previously reported low-cycle fatigue data to the high-cycle regime. A series of unirradiated specimens was tested under high-cycle conditions, but only one irradiated specimen has been tested. Results are encouraging in that an endurance limit appears to have been reached at a strain range of 0.3%.

3.3 The Effect of Preinjected Helium on the Microstructure and Tensile Properties of EBR-11-Irradiated 20%-Cold-Worked Type 316 Stainless Steel (Oak Ridge National Laboratory) 35

Samples of 20%-cold-worked type 316 stainless steel have been irradiated to 8.4 dpa in EBR-II. Irradiation temperatures were 500 and 625°C, and the content of helium (injected near room temperature) was 0, 110, or 200 at. ppm. Samples for all conditions were examined by transmission electron microscopy (TEM), and those irradiated at 625°C were postirradiation tensile tested at the

same temperature. The microstructure of uninjected material shows sparse precipitation of tau and eta phase, a recovered dislocation structure, and no cavities or voids at either 500 or 625°C. Helium preinjection has no significant effect on the dislocation structure at either temperature. Furthermore, preinjection of 110 at. ppm He and irradiation at 500°C produces no resolvable (greater than about 3 nm) distribution of cavities but does reduce slightly eta and tau formation and does enhance Laves formation compared with uninjected material. Preinjection of either 110 or 200 at. ppm He and irradiation at 625°C produces a distribution of about 1 × 10^{22} cavities/m^3, 4 to 5 nm in diameter, on the dislocation structure. Copious precipitation of Laves, eta, and/or tau was observed. These results are compared with the same material after thermal aging or HFIR irradiation at similar temperatures and times. Some microstructural components differ significantly. Tensile testing at 625°C of the uninjected material shows a decrease in strength and increase in elongation compared with unirradiated material. Helium preinjected material generatty shows greater strength but reduced ductility compared with uninjected material.

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Hydrogen charging of Ti-6242s material is complete. Machining of modified-WOL specimens is in progress. No spontaneous hydride precipitation was observed in the material hydrogen charged to 24,100 appm (530 wppm). Room temperature tensile properties were slightly higher for the hydrogen charged specimens than for the as-received specimens.

5.3 Mechanical Property Testing of Unirradiated Path C Alloys (Oak Ridge National Laboratory) 79

Miniature hourglass-shaped fatigue specimens of V—15% Cr—5% Ti have been machined and heat-treated to the desired microstructure of cyclic fatigue testing. Strain-controlled fatigue tests on Nb—1% Zr indicate that the alloy has about the same fatigue resistance at room temperature and at 650°C. Limited data suggest that this alloy has marginally better fatigue properties than does 20%-cold-worked type 316 stainless steel for low strain ranges at 430°C.

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The presence of interstitial hydrogen, in concentrations as low as 1-2 weight ppm, is known to severely affect the fracture behavior of martensitic steels. The embrittling effect increases with lattice concentration of hydrogen and with increases in the yield strength of the alloy in question. The effect of temperature is more complex, however, with a maximum in embrittlement evident near room temperature. This report summarizes the first experiments to evaluate the hydrogen compatibility of HT-9 and to separate the effects of temperature and hydrogen pressure. Tensile tests were performed in air and 15 psi hydrogen at test temperatures of 25, 200, 300 and 400°C and at 25°C in hydrogen pressures of 15, 1000, 2000, 3000 and 5000 psi. The results show that elevated temperatures eliminate any hydrogen effects, consistent with the behavior of other steels having similar strengths. Increasing pressures, however, cause a strong shift in ductility, with an -60% loss in ductility occurring in 5000 psi hydrogen.

7.2 Weldability of HT9: The Autogeneous GTA Process (Sandia National Laboratories) 98

The autogenous GTA process was selected in order to produce a variety of microstructures which will normally be observed in HT9 weldments. Evaluation of the as-welded microstructures indicated that the rapid cooling mtes associated with the weld thermal cycle imparted full martensitic hardness to the fusion zone and portions of the HAZ. At least four distinct regions have been identified within the HAZ; each region could be related to the equilibrium phase diagram of the Fe-12Cr-0.2C alloy system.

7.3 Procurement of National 12 Cr Heat and Evaluation of Welding Procedures for Irradiation Specimens (General Atomic Company) 109

A 14,700 kg (32,500 lb) heat of 12 Cr-1 Mo-0.3 V steel was procured for use by all participants in the National Fusion Ferritic Steel Program. Preliminary analysis indicated that the ingot composition met specifications.

A preliminary evaluation of candidate welding procedures for 12 Cr-1 Mo-0.3 V EBR-11 irradiation weld specimens was completed. The procedure selected for producing the specimens was completed. The procedure selected for producing the specimens was gas-tungsten arc (GTA) with a pre-heat temperature of 93°C.

7.4 **Microstructural and Fatigue Crack Growth Characterization of HT-9 (Hanford Engineering Development Laboratory) . . .** 132

A microstructural characterization of nonirradiated HT-9 has been completed. Selected thermomechanical heat treatments were chosen for study. Heat treatment times for 0.30 to 2.54 mm section thickness were identified by characterizing the austenization behavior of the ferritic alloy. A tempering curve was constructed and complementary transmission election microscopy micrographs taken. Fatigue crack growth and fracture toughness studies were completed on four over-tempered conditions of the alloy. Eased on the microstructural and mechanical properties studied, an optimum thermomechanicat heat treatment for HT-9 was selected.

7.5 Fracture Toughness Tests on HT-9 at Elevated Temperatures (Hanford Engineering Development Laboratory) 156

Single specimen J-integral fracture toughness measurements were rmde at 149°C and 232°C on HT-9 specimens. The electropotentiat technique developed in past work was applied for elevated temperature tests to obtain a calibration curve correlating voltage change to crack extension. Based on this calibration curve, the J versus a curves were generated from single specimens. The test results show that the electropotential technique for single specimen measurements is successful at elevated temperatures, where temperature gradients are of concern. The upper shelf fracture toughness of HT-9 is evaluated as a function of temperature.

7.6 The Fracture Resistance of HT-9 After Irradiation at Elevated Temperature (Naval Research Laboratory) 163

HT-9, a 12 percent Cr martensitic stainless steel of interest for use in ducts for fast reactors and as a first wall material in magnetic fusion reactors, was irradiated in EBR-11 to obtain the first evaluation of fracture toughness changes produced by high temperature-high fluence irradiation in this class of alloy.

Charpy-V notch, precracked Charpy and tensile specimens were used to evaluate the tensile and fracture behavior of this steel in the as-received condition, after 5000 h of aging at 427 and at 538°C and after irradiation to a fluence of 1.1 × 10^{22} n/cm^2 E > 0.1 MeV at 419°C in flowing sodium. The results show some tendency toward temper embrittlement at 538°C and a 108°C shift in the ductile-to-brittle transition after irmdiation at 419°C. Charpy-V energies on the upper shelf are also depressed by the irradiation but maintain adequate levels for the applications intended. Possible mechanisms for the observed response are discussed.

7.7 Environmental Effects on Properties of Ferritic Steels (Argonne National Laboratory) 181

This program is part of a more comprehensive program to investigate the potential of ferritic steels for fusion reactor first-wall blanket structures. Test environments to be investigated include lithium, water, and helium with appropriate impurities as well as candidate solid breeding materials and neutron multipliers. Emphasis will be placed on the combined effect of stress and chemical environment on corrosion and mechanical properties of ferritic steels. Initial tests will focus on the Sandvik Alloy HT-9 and a developmental Fe-9Cr-1Mo alloy.

This program was initiated in the first quarter of FY 1980 with initial phases of the program focused on (1) combined effects of stress and coolant environment on the corrosion behavior and fatigue properties of ferritic steels and (2) the compatibility of ferritic steels with candidate solid tritium-breeding mterials and neutron multipliers, viz., Li_2O, $LiAlO_2$, and Li_2SiO_3.

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8.1 Irradiation Experiment Status and Schedule 186

Bar charts show the irradiation experiments schedule, including experiments completed, in progress, and planned. Experiments are presently under way in the ORR and the HFIR, which are mixed-spectrum reactors, and in the EBR-11, which is a fast reactor.

8.2 ETM Research Materials Inventory (Oak Ridge National Laboratory and McDonnell Douglas Astronautics Company) . . 193

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 materials for the Fusion Reactor Materials Program. This wilt minimize unintended materials variables and provide for economy in procurement and for centralized record-keeping. Initially this inventory is to 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. CORROSION TESTING AND HYDROGEN PERMEATION STUDIES 199

9.1 Hydrogen Dissolution and Permeation Studies of ADIP Program Alloys (Argonne National Laboratory) 200

Efforts to characterize the hydrogen dissolution and permeation characteristics of alloys currently under development in the ADIP Program have continued. Two sets of Ti-6Al-2Sn-4Zr-2Mo-0.08Si plates were loaded with controlled quantities of hydrogen using an existing tensimetric apparatus at ANL. Chemical analyses made at MDAC a one of the two sets confirmed the adequacy of the loading method (540-560 wppm H analyzed versus 500 ± 10 wppm H determined from tensimetric additions). During the course of the loadings, it was found that dehydriding of alloy plates from 500-700 wppm H to lower values) could not be readily achieved without heating the material to well over 600°C. These plates will be used by MDAC to conduct metallurgical and mechanical analyses of hydrogen effects on titanium alloys. In another study, an experiment was conducted to measure the hydrogen permeation characteristics of Fecralloy (Fe-16Cr-5Al-0.3Y) between 190 and 680°C using hydrogen driving pressures in the range from 2 to 1.5 × 10^4 Pa. Comparison of the data from this study with similar results for Fe-Cr alloys having 0 and 2 wt.% Al showed that the aluminum content required to achieve the maximum hydrogen permeation resistance for these alloys in oxidizing environments may be less than 2 wt.% in some cases and could be related to the chromium content in the alloy.

9.2 Vanadium Alloy/Lithium Pumped-Loop Studies (Argonne National Laboratory) 206

The stainless steel-clad vanadium alloy loop with circulating lithium has continued to operate through the second quarter of FY 1960. Test samples of zirconium, vanadium, V-15Cr-5Ti, titanium, and yttrium have been exposed at 600°C to investigate the distribution of nonmetallic elements in lithium/refractory metal systems. The zirconium, titanium, and yttrium exposures are being used to adjust and evaluate the nonmetallic (O, N, H, and C) concentration in lithium. Results of hydrogen analyses of the test samples have been compared with calculated distribution coefficients for hydrogen. Preliminary measurements of the distribution coefficients for nitrogen between vanadium and lithium at 600°C are in good agreement with calculated values.

9.3 Compatibility of 2 1/4 Cr-1 Mo Steel with Static Lithium (Oak Ridge National Laboratory) 213

Tensile specimens of 2 1/4 Cr-1 Mo steel were exposed to static molten lithium at 500°C. After 500 and 1000 h, weight changes were negligible; however, the yield and ultimate tensile strengths of the steel were consistently lower by 4 to 10% than those of specimens thermally aged in argon for the same lengths of time. Analysis of the post-test lithium indicated a transfer of carbon from the steel to the lithium during exposure. Specimens that were normalized and tempered lost less carbon than those that were simply annealed. The carbon concentration of the post-test lithium was greater than predicted by thermodynamic calculations. Therefore, it is believed that the predicted concentrations are in error because the assumption of equilibrium among carbon, molybdenum, and chromium in the steel is not met by the heat treatment conditions used.

9.4 Mass Transfer of Type 316 Stainless Steel and Path B Alloys in Lithium Thermal-Convection Loops (Oak Ridge National Laboratory) 219

Metallographic and surface composition data are reported for the corrosion of ADIP alloys B1 and B3 [58 Fe-24.5 Ni-10 Cr-1 Mo-1 Mn-1.5 Al-3 Ti (wt %) and 55 Fe-30 Ni-12 Cr-2 Nb-1 Mn-2 Ti (wt %), respectively] in lithium flowing between 500 and 300°C. At 500°C both alloys were severely depleted of nickel and exhibited porous near-surface regions. Alloy B3 had a larger volume fraction of porosity. Results on deposition rates vs

exposure time in lithiumtype 316 stainless steel thermal-convection loops are also presented. Deposition mtes in lithium flowing between 600 and 450°C are initially much higher than those measured at later operating times. The decrease in the deposition mte can be correlated with observed changes in the deposition process.