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Alloy Development for Irradiation Performance

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CONTENTS

The nuclear analysis of the hafnium corepiece for the ORR-MFE-4A experiment has been completed. The results of this analysis indicate acceptable helium production to displacement damage ratios over the lifetime (>50 dpa) of the experiment may be obtained using solid aluminum corepieces containing a 0.65-mm-thick hafnium annulus.

The ORR-MFE-4A experiment, described previously, was installed in the Oak Ridge Research Reactor (ORR) a June 10, 1980, and as of September 10, 1981, it had operated for an equivalent 330 d at 30 MW reactor power, with maximum specimen temperatures in each region of 330 and 400°C, respectively. On September 8, 1981, two of the multijunction thermocouples located in the lower region of the capsule, as previously described, indicated sharp drops in temperatures of about 90°C a each. The capsule was removed from the reactor for investigation. Tests have indicated that there are no leaks in the secondary system and no leaks between the thermocouple well and primary system. The possibility of rearranging primary and secondary system boundaries to permit continuation of the capsule irradiation is under investigation.

The ORR-MFE-4B experiment, installed in the ORR on April 23, 1981, is essentially identical to ORR-MFE-4A. Its upper region operates at 600°C, and its lower region operates at 500°C. As of September 30, 1981, it has operated for an equivalent 136 d at 30 MW reactor power.

The HFIR-MFE-RBI, -RB2, and -RB3 experiments are planned for low-temperature irradiation of a variety of specimen types of ferritic steels to approximately 10 and 20 dpa. The final specimen matrices for RB1 and RB2 are given, along with a preliminary matrix for RB3. Also given are details a the alloys included and their conditions. Assembly of the RBI and RB2 capsules is presently under way. Irradiation of the RBI capsule is planned to start in November 1981, and irradiation of RB2 is expected to begin about January 1982.

The HFIR-MFE-T3 experimental capsule is described. This experiment consists of miniature Charpy V-notch specimens of 12 Cr-1 MoVW and 12 Cr-1 MoVW-2 Ni alloys. The

and compared after thermal aging, HFIR irradiation at 55 to 750°C at fluences producing up to 16 dpa and 1020 at. ppm He, and EBR-11 irradiation to 8.4 and 36 dpa at 500 to 630°C. The CW 316 shows dislocation recovery on aging in the range 550 to 700°C, with recovery increasing as temperatures increase. By comparison, CW 316 + Ti exhibits MC formation and resists dislocation recovery for 4400 h at 700°C. Dislocation recovery and precipitation are uncoupled in CW 316 after EBR-11 irradiation. Recovery is enhanced and precipitation is retarded. The DO heat of CW 316 does not form Frank loops in the 500 to 630°C range. Network dislocation concentrations tend toward a steady value of 1 to 3 × $10^{14} m/m^3$, with only slight temperature and fluence variations. During HFIR irradiation, CW DO heat 316 shows dislocation recovery after 7 to 10 dpa, with moderate temperature dependence from 55 to 450°C and stronger temperature dependence above 450°C. Between 55 and 550°C the dislocation recovery is strongly coupled to other microstructural features. Frank loops are found for irradiation at 450°C and below, cavities at 350°C and above, and precipitation at 450°C and above. The dislocation structure recovers to similar values in HEIR and EBR-II at 500 to 630°C.

The dislocation microstructure in CW 316 + Ti is similar to CW 316 after HFIR irradiation from 55 to 750°C, but the coupling of network recovery, Frank loop behavior, and other microstructural phenomena is different from those in CW 316. The greatest differences reflect MC dislocation pinning and reduced phase instability. Dislocation concentrations are 3 to 5 times higher in the CW 316 + Ti than in the CW 316.

Equations describing cavity volume fraction swelling are developed for 20%-cold-worked type 316 stainless steel (CW 316) irradiated in HFIR. These equations are based on the physical phenomena observed in the microstructure, such as matrix void swelling, matrix bubble swelling, grain boundary cavity swelling, and precipitate-assisted void swelling. The temperature and fluence dependence of each component is considered, and microstructural information is interpolated to provide a description of the swelling behavior within the limits of the data set (up to 50–60 dpa). The data base is for the DO heat of CW 316. Some facets of the swelling behavior, such as void attachment to eta (M_6C) phase particles at lower temperature, may vary between heats of type 316 stainless steel. Other components, such as bubbles in the matrix and grain boundary cavities, may be more general. The expression of *total swelling as the sum of distinct, microstructurally related components allows flexibility in describing other heats of steel having generally similar behavior but with some differences in precipitate response, for example. It also permits separate comparison of components of the swelling, such as bubbles or void formation, with these same phenomena observed under different irradiation conditions. Comparison of the overall swelling in HFIR with that observed in EBR-11 indicates substantial differences in the temperature and fluence dependence of swelling in these reactors. The microstructural information shows that the differences are due to the helium generation mte and its effect a various mechanisms responsible for development of the cavity microstructure. An understanding of these differences and development of physically based models that predict both EBR-11 and HFIR data will be necessary to project the swelling behavior in a fusion reactor.*

Samples of 20% CW 316 SS were sectioned from various axial locations along an irradiated EBR-11 duct. The bend samples were fabricated by Tungsten-Inert-Gas (TIG) welding two tabs together along the width. Bending was accomplished by centrally loading the root side of the weld white both ends of the specimen were supported by pins. The effects of sample fluence level, test temperature, and deflection rate on strength and ductility were investigated. A preliminary evaluation indicates the ductility of the welded material is much greater than expected. Helium produced in the metal during reactor service does not appear to cause embrittlement of the weld zone.

No contributions.

Creep/stress-rupture tests were performed a sheet specimens of the three Path C vanadium Scoping Alloys in the temperature range 650 to 800°C. As expected, the V-15Cr-5Ti was strongest in creep, followed by VANSTAR-7 and V-20Ti. These tests represented over 12,000 hours of testing in the ultra-high vacuum creep test stands. In addition to the creep tests, a series of controlled nonmetallic contamination exposures were carried out at 800°C.

surprising since, in general, higher strength microstructures are more severely degraded by hydrogen. Current efforts are aimed at understanding these results, and assessing their impact a the applicability of HT-9 as a first wall material.

A review of the current literature, industrial experience in both the U.S. and Europe, and the results of research performed under the ADIP Path E program has resulted in a number of observations and recommendations regarding the weldability and long term integrity of HT-9. In the opinion of the authors, the weldability characteristics of HT-9 does not preclude this alloy from consideration as a first wall/blanket material for fusion machines. Indeed, weldability observations on this alloy, to date, are encouraging. However, although the transformation and tempering response of the fusion zone and heat-affected zone (HAZ) has been well characterized, optimization of the welding process and process parameters will be necessary in order to successfully fabricate the first wall modules. In particular, there are several factors which may affect weld joint integrity and must be studied to further define the weldability of HT-9. Thesse include evaluation of the effects of hard triaxial restraint, discontinuities and defects, delay time prior to post-weld heat treatment and horizontal and vertical weld positions. In addition. the weldability and weld integrity of product forms pertinent to first wall/blanket structures must be studied. Evaluation of these aspects is the next logical step in determining whether reliable weld joints of HT-9 can be fabricated in the shop or field. Both the gas tungsten-arc (GTA) and laser welding processes have been demonstrated as suitable techniques for joining HT-9, although the choice of laser welding as a primary or secondary joining process wilt require considerable process control to ensure reliable welded joints. Finally, in order to optimize both the welding process and the postweld heat treatment (PWHT) which will be required it is necessary to determine the minimum mechanical properties necessary to ensure the fabrication and safe operation of fusion reactor devices.

The second part of this report to be published in a future ADIP quarterly will focus on areas of future research which will be necessary to qualify HT9 weldments for use in irradiation and hydrogen environments.

The electropotential technique has been applied to determine J_{1c} on single specimens of HT9. The technique was extended to 9Cr-1Mo in this work. Fracture toughness tests were performed on unirradiated 9Cr-1Mo specimens at 25, 232 and 427°C, and on HT9 specimens at 25 and 232°C for comparison. Continuous crack extension measurements and J versus Δa curves were obtained through the use of a semi-empirical expression in terms of V/V_o and a/a_o. The analysis of tests results shows that for HT9, the single specimen method agrees well with the multi-specimen method in determining J_{1c}, however, there is noticeably larger (order 15%) uncertainty for 9Cr-1Mo tested at 427°C. Alloy 9Cr-1Mo shows less variation in the temperature dependence of J_{1c} and a higher resistance to crack propagation than HT9.

TEM specimens of the ferritic alloys HT-9, 9Cr-1Mo and 2 1/4Cr-1Mo have been prepared for inclusion in the HFIR-MFE-RBI irradiation. The specimen matrix encompasses all the conditions being irradiated in the EBR-11 AD-2 test. Additionally, the matrix includes specimens of HT-9 weld fusion zones and simulated heat-affected zones to study the microstructural response of weldments.

A miniature charpy v-notch (CVN) type impact specimen geometry has been selected and two instrumented drop towers have been purchased from Effects Technology, Incorporated (ETI) and received at HEDL. Impact testing of unirradiated HT9 miniature CVN specimens has been performed at temperatures of -100 to +100°C. The ductile to brittle transition temperature obtained is in excellent agreement with data obtained using full size HT9 CVN specimens. Dynamic fracture toughness data can also be obtained. However, improved instrumentation will be required to obtain dynamic fracture toughness data outside the lower shelf region.

Charpy specimens of HT-9 were given a heat treatment 5 minutes at 1038°C followed by a water quench, then 1 hour at 760°C followed by air cooling. Surface cracks were *found in all three specimens following heat treatment. Charpy tests were performed on these HT-9 specimens to determine the effect of water quenching relative to air cooling. A Charpy test at 23°C (upper shelf behavior) showed the water-quenched HT-9 to be 1.70 times as tough as the HT-9 air cooled. The tests performed at −59°C (lower shelf behavior) showed a similar increase in toughness (up to 1.71 times). The consequence of a water quench is therefore found to be a large increase in upper shelf energy which results in an effective DBTT shift of ~40°C but only a small change in the lower shelf behavior.*

The effect of variations in the heat treatment of 9 Cr-1 MoVNb and 12 Cr-1 MoVW have been evaluated. Dissolution of carbides during austenitization was found to be somewhat faster in 9 Cr-1 MoVNb than in 12 Cr-1 MoVW. The effect of cooling rate after austenitization was strongly dependent a the austenitization time and temperature. The 9 Cr-1 MoVNb alloy structure was found to be more sensitive to cooling rate than the structure of 12 Cr-1 MoVW. Under some circumstances, furnace cooling of 9 Cr-1 MoVNb after austenitizing resulted in a ferrite plus carbide structure rather than a martensite lath structure, and in addition the carbide was not the same as that formed during tempering. The precipitation reactions in both alloys are essentially complete after tempering 1 h at 650°C. In 12 Cr-1 MoVW the principal carbides are chromium-iron rich $M_{23}C_6$ and vanadium rich MC, whereas those in 9 Cr-1 MoVNb were chromium-iron rich $M_{23}C_6$ and niobium-vanadium rich MC.

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

During the present reporting period, irradiated specimens of 2 1/4 Cr-1 Mo steel in the normalized-and-tempered and isothermally annealed conditions were tensile tested at room temperature and 300°C. The yield strength and ultimate tensile strength of the irradiated samples displayed considerable hardening over the unirradiated condition.

alloys or to cause sufficient attack of the specimens exposed at 300 and 400°C. It appears that, for ferrous alloys, the application of Pb–17 at. % Li as a semistagnant breeding fluid in a fusion reactor m y be limited to temperatures of 400°C or less. Containment alloys of low nickel and chromium activities would be preferable.

Weight loss data are reported for the long-range-ordered (LRO) alloy Fe-31.8 Ni–22.5 V–0.4 Ti (wt %) exposed to lithium in type 316 stainless steel thermal-convection loops (TCLs) for up to 5000 h at 600°C. Very high corrosion mtes were measured and extensive corrosive attack was observed. The exposed surfaces were depleted in nickel and correspondingly enriched in iron and vanadium. Another lithium-type 316 stainless steel TCL was used to study the dependence of the dissolution rate of type 316 stainless steel in flowing lithium on temperature. The observed temperature dependence was consistent with an apparent overall activation energy of 160 kJ/mol (38 kcal/mol). This is a higher activation energy than has been measured in earlier tests and this difference indicates problems with reproducibility of activation energies measured in different experiments.

Several constant-stress compatibility tests and continuous-cycle fatigue tests have been conducted on HT-9 alloy and Type 304 stainless steel at 755 K in a flowing lithium environment. The results indicate that for applied stresses below the yield stress of the material, the corrosion behavior of HT-9 alloy and Type 304 stainless steel is independent of stress. The fatigue properties of these materials are strongly influenced by the concentration of nitrogen in lithium. For HT-9 alloy, the fatigue life in lithium containing 100–200 wppm nitrogen is a factor of 2 to 5 greater than that in lithium with 1000–5000 wppm nitrogen. In tow-nitrogen lithium, fatigue life is also independent of strain rate. The lower fatigue lives observed in high-nitrogen lithium m y be attributed to corrosion. Fatigue tests on lithium-exposed specimens are in progress to investigate the long-term environmental effects.

Construction of a forced-flow lead-lithium loop is in progress. Tests have been formulated to investigate the combined effects of stress and environment on the corrosion and mechanical properties of structural mterials.

The compatibility of solid Li_2O, $LiAlO_2$, and Li_2SiO_3 breeding materials with several commercial alloys has been investigated at 873 and 973 K. The results show that Li_2O is the most reactive and $LiAlO_2$ is the least reactive of the three breeding materials. The reaction scales on alloys exposed with Li_2O ceramic contain Li_5FeO_4 and $LiCrO_2$ compounds. The formation of those compounds may explain the greater interaction between the alloys and Li_2O material. Compatibility tests at 773 K are in progress. A compatibility-test facility is being constructed to study the alloy/ceramic interactions in a flowing helium environment containing known amounts of moisture.