DOE/ER-0313/1

Fusion Reactor Materials

Semiannual Progress Report
for Period Ending
September 30, 1986

U. S. Department of Energy
Office of Fusion Energy

DOE/ER-0313/1
Distribution
Categories
UC20, 20c

FUSION REACTOR MATERIALS SEMIANNUAL PROGRESS REPORT FOR THE PERIOD ENDING SEPTEMBER 30, 1986

Date Published: September 1987

Prepared for
DOE Office of Fusion Energy

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under Contract DE-AC05-84OR21400

FOREWORD

This is the first in a series of semiannual technical progress reports on fusion reactor materials. This report combines research and development activities which were previously reported separately in the following technical progress reports:

- Alloy Development for Irradiation Performance
- Damage Analysis and Fundamental Studies
- Special Purpose Materials

These activities are concerned principally with the effects of the neutronic and chemical environment on the properties and performance of reactor materials; together they form one element of the overall materials program being conducted in support of the Magnetic Fusion Energy Program of the U.S. Department of Energy. The other major element of the program is concerned with the interactions between reactor materials and the plasma and is reported separately.

The Fusion Reactor Materials Program is a national effort involving several national laboratories, universities, and industries. The purpose of this series of reports is to provide a working technical record for the use of the program participants, and to provide a means of communicating the efforts of materials scientists to the rest of the fusion community, both nationally and worldwide.

This report has been compiled and edited under the guidance of A. F. Rowcliffe, Oak Ridge National Laboratory, and D. G. Doran, Battelle-Pacific Northwest Laboratory. Their efforts, the work of the publications staff in the Metals and Ceramics Division at ORNL, and the many persons who made technical contributions are gratefully acknowledged. T. C. Reuther, Reactor Technologies Branch, has responsibility within DOE for the programs reported on in this document.

G. M. Haas, Chief
Reactor Technologies Branch
Office of Fusion Energy

CONTENTS

The Workshop on Mechanical Properties of Fusion Reactor Materials was held in conjunction with the Second International Conference on Fusion Reactor Materials to encourage international representation in this workshop series. The meeting was held in Chicago, Illinois in April 1986, with participants from Japan, Europe, and the United States. The topics covered were: 1) flow processes, 2) time dependent crack growth, 3) time independent crack growth/brittle fracture, and 4) environment assisted crack growth. The background, status, and recommended research are summarized in this report for each of the workshop topics.

Internal hydrogen concentrations of 4 appm were found to have a significant effect on crack growth rate in HT-9 tested at room temperature. This effect was most pronounced at ΔK's less than about 15 MPa-m$^{1/2}$ for tests conducted at a load ratio of 0.5. At a load ratio of 0.05 internal hydrogen had very little effect on the fatigue crack growth rate. Hydrogen induced an intergranular fracture mode which increased in percentage of the total fracture with decreasing crack velocities at decreasing ΔK's. Hydrogen has been shown to induce intergranular fracture of HT-9 in precharged and dynamic tensile tests and subcritical crack growth tests at cathodic potentials. The fatigue crack growth rate of both the hydrogen charged and uncharged material was a function of the cyclic frequency between frequencies of 0.5 and 5 Hz with the fatigue crack growth rate increasing with decreasing frequency or increasing period. There is some evidence that the as-received hydrogen concentration of 1 ppm affected the crack growth rate. The crack velocity was also dependent on the load ratio with hydrogen having a large effect on the fatigue crack growth rate at a load ratio of 0.5 and a small effect at a load ratio of 0.05. The observed results could have significance to fusion reactor design if they are shown to persist to reactor temperatures. A combination of modeling and experiment will be used to help answer this question.

The microstructure of copper and three single-phase copper alloys (Cu-5%Al, Cu-5%Mn, Cu-5%Ni) has been examined following a room-temperature 14-MeV neutron irradiation up to a maximum fluence of 2 x 10^{21} n/m^2 (~10^{-3} dpa). The irradiation produced small defect clusters, many of which were resolvable as stacking fault tetrahedra (SFT) or triangle loops. Alloying did not affect the overall cluster density or size distribution, but it did cause a significant reduction in the density of SFT and triangle loops. The overall defect cluster size was independent of fluence with a mean and most probably diameter of ~2.7 and ~1.7 nm, respectively. There was no evidence of cascade overlap in this study. Cascade overlap effects are estimated to become significant for fluences ~10^{23} n/m^2. The overall defect cluster density was found to vary as $N \sim \sqrt{\phi t}$ with a short incubation period for all four metals. Reexamination of published data indicates that this relationship may be broadly applicable over the irradiation temperature range 80 to 400 K. Logarithmic plots of the cluster density versus fluence produced anomalous curvature due to the influence of the incubation period.

The status of the OWR/RTNS-II Low Exposure Spectral Effects Experiment is reviewed. The originally planned irradiations in RTNS-II and the Omega West Reactor at 90°C and 290°C have been completed, and additional irradiations in RTNS-II at 200°C and 450°C have also been carried out. Tensile specimens from each of the irradiations have been tested. The effects of irradiation temperature on the change in yield stress as a function of 14 MeV neutron fluence are reported here for annealed pure copper, annealed 316 stainless steel and HT9 ferritic steel. In copper the effect of increasing the irradiation temperature is to increase the minimum fluence at which a measurable change in mechanical properties takes place. The temperature effects are somewhat smaller in

dimensional nature of the decomposition process. In low dose neutron-irradiated Fe-Ni-Cr specimens, the use of high electropolishing rates during preparation of microscopy foils leads to a preferential attack of those regions which are low in nickel. In high dose ion-bombarded specimens of Fe-35Ni, the absence of chromium leads to formation of cellular martensite in the nickel-poor regions upon cooling to room temperature.

The scale of the fluctuations at temperatures >600°C is comparable to that found in the Santa Catharina meteorite, which is roughly Fe-35%Ni in composition. This and other non-radiation data support the proposal that the Fe-Ni system in the absence of irradiation tends to spinodally decompose in the invar regime but at a very sluggish rate. Thus it appears that radiation accelerates rather than induces the decomposition of Fe-Ni and Fe-Ni-Cr invar alloys.

Swelling data from both ion and neutron irradiations of Fe-Cr-Ni alloys are analyzed in terms of the critical radius concept for bubble-to-void conversion. It is shown that while the dependence of critical radius on temperature, displacement rate and helium can explain many features of the data, one must also consider the dependence of vacancy diffusion on nickel content in order to be able to explain the compositional dependence of void nucleation.

A recent ion bombardment study has called into question a fundamental assumption used to design a Ni^{59} isotopic tailoring experiment. This assumption involves the synergistic roles of phosphorus and helium in void nucleation and therefore the assumption was tested by analysis of the results of an earlier irradiation experiment. Neutron irradiation of Fe-25Ni-15Cr with three different phosphorus levels was found to yield density changes which support the design assumption. Based on the role proposed for phosphorus an alternative explanation is also offered to describe the results of the ion bombardment experiments may not be suitable for the simulation of the influence of phosphorus on neutron-induced microstructural development.

Data on the neutron and ion-induced swelling of pure nickel and nickel-chromium alloys are reviewed to test the hypothesis that long or short range ordering will reduce swelling. The concept of order-induced swelling suppression appears to be valid, but the influence of chromium on ordering and swelling of nickel-chromium alloys is complicated by the action of at least one other as-yet-undetermined mechanism. The swelling rate of pure nickel depends on temperature and metal purity, reaching ~1%/dpa at low temperatures and high purity. Nickel also saturates in swelling at a pace dictated by purity, cold-work level and temperature, while Ni-Cr alloys do not appear to be subject to saturation.

Nickel samples with various helium and oxygen concentrations have been irradiated with 14 MeV Ni ions at 500°C to a fluence of 8×10^{19} ions/m² (2 dpa at a 1-µm depth). Helium atoms with energy varying from 200 to 700 keV were pre-injected at room temperature. The samples with low oxygen content were obtained by a hydrogen reduction treatment and high vacuum outgassing. The density and the average diameter of voids were determined by TEM examination of cross-section specimens.

The residual oxygen plays an important role in promoting void formation. Lowering the oxygen content from 180 appm to 75 appm reduces void density and increases void size remarkably.

Small amounts of helium (10 appm) enhanced the void nucleation remarkably in both high (180 appm) and low (75 appm) oxygen content samples, while larger amounts of helium (30 appm) reduced the observable void density in the high oxygen content sample.

Surface energy values lower than those determined experimentally are often utilized in theories of void nucleation and growth in metals. Utilization of established surface energy values generally predicts no swelling in the absence of

Solution-annealed (SA) and 20%-cold-worked (20% CW) type 316 stainless steels were irradiated in HFIR up to 33 to 36 dpa and 2250 to 2335 appm He at irradiation temperatures of 300 and 400°C. Small bubbles (1.5 to 4.5 nm in diameter) were uniformly dispersed throughout the matrix at concentrations in the range 2 to 4 x 10^{23} m^{-3}. Swelling was very low (below 0.2%) in both materials. In SA materials, cavity size rapidly increased while the number density decreased at irradiation temperatures of 500°C and above. Swelling appeared to be a maximum at 500°C (>1%). Most of the cavities were voids at 600°C. On the other hand, in 20% CW specimens, cavities were much smaller, with diameters of 6 and 9 nm at 500 and 600°C, respectively. The cavity number density at both 500 and 600°C (~1 x 10^{22} m^{-3}) was about one order less than at 400°C. Swelling slightly increased as irradiation temperature increased, peaking at 600°C (0.3%). Inhibition of swelling by cold working was more effective at temperatures above 500°C.

Tensile properties were determined for six Fe-Mn-Cr-C alloys that were used previously to determine the austenite-stable region in that system. The steels were tested in the solution-annealed and 20%-cold-worked conditions. When the results were compared with type 316 stainless steel, the average behavior of the five steels compared favorably with the tensile behavior of type 316 stainless steel.

Initial phases in the production of V-Cr-Ti alloys have been completed. All steps in the process flow sheet (1) have been activated on production of the V-15Cr-5Ti ternary alloy. Sufficient material was processed to generate a 9.65 cm (3.8") diameter cast ingot weighing 13.1 kg (29 pounds). Subsequent hot extrusion provided a change in geometry to the billet to remove the cast structure and produce a rectangular shape to initiate hot rolling procedures. This extrusion has been subdivided and is currently passing through final processing operations, which include hot rolling, warm rolling and cold finishing steps. Flat stock is now being accumulated, while characterization work such as chemistry, non-destructive testing and metallography is on-going. Plans for the fabrication of the V-10Cr-5Ti alloy have been defined and the initial alloy consolidation is under way.

In addition, this effort provides for correlation of complimentary fabrication work on V-12Cr-5Ti and V-10Cr-10Ti alloys at Teledyne Wah Chang Albany.

The V-3Ti-1Si, V-5Ti, V-20Ti, and V-15Cr-5Ti alloys were irradiated at 650°C with 4.0-MeV $^{51}V^{++}$ ions to 50 dpa. Voids were not visible in the irradiated alloys. Coherent precipitates were produced in the irradiated V-5Ti and V-15Cr-5Ti alloys. The precipitates were noncoherent with the matrix in the V-20Ti alloy and partially coherent in the V-3Ti-1Si alloy. The formation of coherent precipitates may contribute to the greater susceptibility of the V-15Cr-5Ti alloy, in comparison to the V-20Ti and V-3Ti-1Si alloys, to irradiation hardening and loss of ductility after neutron irradiation. A test matrix for irradiation of V-base alloys in the FFTF reactor during cycle 9 is presented.

Vanadium-20% titanium tubes, pressurized to stresses of 34 and 39 MPa, were irradiated in the Experimental Breeder Reactor (EBR-II) at 700°C to a fluence of 3.9 x 10^{26} n/m^2, corresponding to a displacement damage level of 22 dpa. Sections of the tubes were injected with 15 appm He prior to irradiation to determine the effect of helium on the microstructural and creep response of this

initial study of low activation ferritic steels in thermally convective lithium at 500°C was completed. Results showed low weight losses typical of standard ferritic steels exposed under similar conditions. Steels containing a significant concentration of manganese appear to be slightly less corrosion resistant than one containing only 0.02% wt % of this element.

During the current reporting period, long-term (10,000 h) baseline thermal convection loop experiments with austenitic and ferritic steels exposed to Pb-17 at. % Li were completed. The 500°C data confirmed the aggressiveness of the lead-lithium enviroment. Surface analysis revealed uniform attack of 12Cr-1MoVW steel with minimal change in surface composition. In contrast, the type 316 stainless steel suffered irregular attack and preferential dissolution of nickel and chromium. These observations are in accord with a model that predicts such nonuniform attack under conditions of selective leaching. Mass transfer profiles revealed that, in the case of type 316 stainless steel, the maximum deposition was not at the coldest point in the loop. Such behavior can be attributed to the product of thermodynamic and kinetic factors that vary oppositely with respect to temperature.

Corrosion data are presented for several ferritic steels and the V-15Cr-5Ti alloy in a flowing lithium environment. The dissolution rates of low-activation ferritic steels and V-15Cr-5Ti alloy are compared with those for HT-9 and Fe-9Cr-1Mo steels. The influence of nitrogen content in lithium on the corrosion behavior of these alloys is discussed.

Corrosion data have been obtained on low-activation ferritic steels in flowing Pb-17 at. % Li environment at temperatures of 482 and 371°C. The results are compared with the dissolution behavior of ferritic HT-9 and Fe-9Cr-1Mo steels and the austenitic Type 316 stainless steel.

Lithium-containing ceramic tritium-breeder materials have been envisaged to be deployed within the blanket region of a fusion reactor in several possible configurations. One of these is the sphere-pac configuration. For this configuration an important material parameter is its thermal conductivity (K_{sp}). It is well known that K_{sp} demonstrates rather complex behavior as a function of temperature, gas pressure, gas composition, particle size, and packing fraction. The interrelationship of these parameters has been satisfactorily accounted for with a hierarchical effective media theory (HEMT). For tritium self sufficiency, most lithium ceramic breeder materials would require the presence of a neutron-multiplier (e.g., Be or BeO). Here, the influence of configuration on K_{sp} (i.e., how one put the different solid components together in the sphere-pac bed) becomes important. Using a generalized HEMT (i.e., a model with capability to describe systems with more than one solid material component), we have analyzed in detail the configurational dependence of K_{sp} for sphere-pac beds composed of lithium ceramic/BeO microspheres. Substantial improvements in K_{sp} can be achieved if a configuration of lithium ceramic spheres coated with BeO is chosen. Increases in K_{sp} would lead to enhanced mechanical and thermal performance of the breeder materials.

The BEATRIX experiment is an IEA-sponsored effort that involves the exchange of solid breeder materials and shared irradiation testing among research groups in several countries. The materials will be tested in both closed capsules (to evaluate material lifetime) and opened capsules (to evaluate purge-flow tritium recovery). Pre- and post-irradiation measurement of thermophysical and mechanical properties will also be carried out.

Experimental measurements are being made of surface adsorption of H_2O on $LiAlO_2$, the solubility of hydroxide in $LiAlO_2$, and the kinetics of release of H_2O from $LiAlO_2$. Up to about 500°C, evolution of H_2O is first order in dissolved protons (hydroxide). At higher temperatures, the reaction appears to shift to second order. Solubility of hydroxide appears to decrease with increasing temperature. A second condensed phase can appear at about 315°C for a partial pressure of H_2O of 550 vppm. The critical partial pressure of H_2O to form a LiOH-rich second phase can be similar for all breeders. Two different kinds of lattice sites appear to be involved in hydroxide dissolution. Surface adsorption of molecular oxygen or hydrogen can be understood to influence tritium release rates markedly; the thermodynamic and kinetic effects of these gases on the release rates operate in the same direction. "Tritium" diffusion is to be identified with triton diffusion.

The preliminary measurements of mechanical properties of lithium oxide were completed. Several batches of lithium zirconate (Li_2ZrO_3) powder were synthesized and sent to Hanford Engineering and Development Laboratory to be fabricated into FUBR-1B replacement capsules. Lithium oxide powder was prepared and the fabrication of ring-shaped Li_2O samples was initiated for the CRITIC experiment. Mechanical properties tests were initiated on $LiAlO_2$ and Li_2ZrO_3. Approximately 4000 kg of sintered lithium carbonate blocks were prepared and sent to Japan for neutronics cross-section tests.

A vacuum apparatus was designed and constructed for the rapid measurement of retained helium and tritium in lithium ceramics. The apparatus eliminated the limitations and errors associated with the acid dissolution technique and the previous vacuum annealing technique (below the melting point) and allowed more accurate and less expensive analysis techniques.

Tritium retention in Li_2ZrO_3 was significantly less than in the other ceramics. Tritium retention appears to possess a proportional dependence to burnup. A review of available models reveals that none fully described the absolute magnitude or the relationship of retention to temperature or burnup so that a model which considers irradation effects is desired.

An analysis method was developed which allows the transient response of in situ tritium recovery experiments after temperature change steps to be used in calculation of diffusion coefficients. The papers by Kurasawa et al. on the conclusions reached from this work are available in the open literature and will not be reproduced be here.

The first insertion of two subassemblies has completed its irradiation in December 1986. This irradiation exposed Li_2O and $LiAlO_2$ to not only high temperatures but also large temperature gradients which are expected in fusion blankets. In addition, it included other materials such as Li_2ZrO_3, Li_8ZrO_6, Li_4SiO_4 and $LiAlO_2$ (spheres and large grain size) some of which will go to high burnups.

The second insertion will contain lithium ceramics from Saclay, France; Casaccia, Italy; Karlsruhe, Federal Republic of Germany; Springfield Laboratories, England; and JAERI, Japan.

Lithium was transported within the FURB-1A capsules which contained Li_2O and Li_4SiO_4. The temperature and lithium burnup dependence, along with the absolute magnitude of this transport, suggest that it was caused by the formation of LiOT gas above the Li_2O pellet. Although transport in Li_2O blanket designs with high temperatuare purge channels can produce extensive material transport, the transport within the blanket may be limited by the localized burnup of the lithium.

**Possible mechanisms for the doubled dielectric loss include electromagnetically-induced vibration of dislocation entities, and interactions with point defects produced by displacement and transmutation events. Any future specification for a useable fusion ceramic at ECRH or lower frequencies (as for ICRH) will have to include more than a tabulation of desired property values, since compositional, microstructural, and processing variables are also important. These include the relative concentrations of major impurities, firing temperature, and cooling rates after firing.*

An integrated approach has been developed for rapid screening of the influence of component variables on the performance of electrical insulators required for superconducting magnets in magnetic fusion energy systems. It incorporates an efficient method of specimen production in the form of 3.2-mm (0.125-in) diameter rods. Test methods include short-beam shear, fracture strength (G_{Ic}), and strain-controlled torsion. The torsion test induces failure between the fiber and matrix, which is expected to be the dominant failure mode induced by cryogenic irradiation. In addition to providing quantitative data on the modulus of rupture and of rigidity, the strain control feature facilitates analysis of the stress-displacement curve in the region where damage is occurring, providing useful information on how the various component and irradiation parameters are influencing the failure mode. The torsional test facility is easily constructed, provides rapid specimen turnaround, and has a low consumption of cryogens. A specimen subjected to a torsion test may be subsequently tested by the short-beam and fracture strength methods, enabling five tests to be performed on the same specimen. Glass-fiber reinforced specimens having three types of epoxy matrix and one type of polyimide matrix have been produced and submitted to ORNL for irradiation in the NLTNIF facility, after which they will be returned to NBS for testing by these methods.