

**DOE-ER-0313/40
Distribution
Categories
UC-423, -424**

**FUSION MATERIALS
SEMIANNUAL PROGRESS REPORT
FOR THE PERIOD ENDING
June 30, 2006**

**Prepared for
DOE Office of Fusion Energy Sciences
(AT 60 20 10 0)**

DATE PUBLISHED: September 2006

**Prepared for
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
Managed by
UT-Battelle, LLC
For the
U.S. DEPARTMENT OF ENERGY**

FOREWORD

This is the fortieth in a series of semiannual technical progress reports on fusion materials science activities supported by the Fusion Energy Sciences Program of the U.S. Department of Energy. This report focuses on research addressing the effects on materials properties and performance from exposure to the neutronic, thermal, and chemical environments anticipated in the chambers of fusion experiments and energy systems. This research is a major element of the national effort to establish the materials knowledge base of an economically and environmentally attractive fusion energy source. Research activities on issues related to the interaction of materials with plasmas are reported separately.

The results reported are the product of a national effort involving a number of national laboratories and universities. A large fraction of this work, particularly in relation to fission reactor irradiations, is carried out collaboratively with partners in Japan, Russia, and the European Union. The purpose of this series of reports is to provide a working technical record for the use of program participants, and to provide a means of communicating the efforts of fusion materials scientists to the broader fusion community, both nationally and worldwide.

This report has been compiled and edited under the guidance of R. L. Klueh and Teresa Roe, Oak Ridge National Laboratory. Their efforts, and the efforts of the many persons who made technical contributions, are gratefully acknowledged.

G. R. Nardella
Research Division
Office of Fusion Energy Sciences

TABLE OF CONTENTS

1.0	<i>VANADIUM ALLOYS</i>	1
1.1	PROGRESS IN CONSTRUCTION OF A V-4CR-4TI THERMAL CONVECTION LOOP AND TEST FACILITY—B. A. Pint, S. J. Pawel, and J. L. Moser (Oak Ridge National Laboratory)	2
	A vacuum chamber and ancillary equipment are being assembled for the experiment. A loop design has been formalized and V-4Cr-4Ti tubing has been received and examined prior to fabrication of the loop. Specimens and wire for the hot and cold leg have been fabricated. Specimens have been prepared for two layer MHD coatings at Lawrence Livermore National Laboratory.	
2.0	<i>CERAMIC COMPOSITE MATERIALS</i>	6
2.1	IRRADIATION CREEP OF CHEMICALLY VAPOR DEPOSITED SILICON CARBIDE AS ESTIMATED BY BEND STRESS RELAXATION METHOD—Y. Katoh, L. L. Snead, S. Kondo (Oak Ridge National Laboratory), T. Hinoki, A. Kohyama (Kyoto University)	7
	Bend stress relaxation technique was successfully applied for the study on irradiation creep of chemically vapor-deposited beta-phase silicon carbide (SiC) ceramics and vapor-infiltrated composites. Samples machined into thin strips were held within a curved gap in the silicon carbide fixture, and irradiated in High Flux Isotope Reactor at Oak Ridge National Laboratory and Japan Materials Test Reactor at Japan Atomic Energy Agency to the maximum neutron fluences of 0.74×10^{25} n/m ² ($E > 0.1$ MeV) at 400–1030°C. Irradiation creep strain for SiC exhibited weak temperature dependence in the temperature range studied. The creep strain appeared highly non-linear to neutron fluence due to the early domination of the initial transient irradiation creep. The transient creep was speculated to have caused by the rapid development of defect clusters and the structural relaxation of as-grown defects during early stages of irradiation damage accumulation. Steady-state irradiation creep compliance of SiC was conservatively estimated to be $2\text{--}3 \times 10^{-32}$ (MPa-n/m ²) ⁻¹ at ~600–~1,100°C. The observed smaller irradiation creep strains for the monocrystalline SiC and the uni-directional composites than for the polycrystalline SiC were attributed to differences in the transient creep strain.	
3.0	<i>FERRITIC/MARTENSITIC STEELS AND ODS STEELS</i>	16
3.1	EVALUATION OF FRACTURE TOUGHNESS MASTER CURVE SHIFT OF JMTR IRRADIATED F82H USING SMALL SPECIMENS—T. Yamamoto, G. R. Odette, D. Gragg, W. J. Yang (University of California, Santa Barbara), H. Kurishita, H. Matsui, M. Narui, M. Yamazaki (Tohoku University, Japan)	17

Small to ultra-small 1/3 size pre-cracked Charpy and 1.67 x 1.67 x 9.2 mm deformation and fracture minibeam (DFMB) specimens of the F82H IEA heat were irradiated to 0.02 and 0.12 dpa at 290°C in the Japanese Materials Test Reactor. Nominal cleavage transition temperature shifts, based on the measured toughness, $K_{Jm}(T)$, data (ΔT_m) as well as reference temperature shifts (ΔT_o) found after size-adjusting the $K_{Jm}(T)$ data yielded $\Delta T_{m/o} \approx 27 \pm 10$ and 44 ± 10 at the two doses, respectively. Using measured yield stress changes ($\Delta \sigma_y$), the $C_o = \Delta T_o / \Delta \sigma_y = 0.58 \pm 0.14$ at 0.12 dpa, is in good agreement with data in the literature. The dynamic transition temperature shift, ΔT_d , derived from DFMB tests, was $\approx 30 \pm 20^\circ\text{C}$ at 0.1 dpa, also in good agreement with the estimated ΔT_o shifts. The ΔT_d and ΔT_o are also in excellent agreement with a $\Delta T_o = C_o \Delta \sigma_y(\text{dpa}, T_i)$ hardening-shift model, where the $\Delta \sigma_y(\text{dpa}, T_i)$ was found by fitting a large database on tensile properties.

- 3.2 EFFECTS OF CONSOLIDATION TEMPERATURE, STRENGTH AND MICROSTRUCTURE ON FRACTURE TOUGHNESS OF NANOSTRUCTURED FERRITIC ALLOYS—P. Miao, G. R. Odette, T. Yamamoto (University of California, Santa Barbara), M. Alinger (University of California, Santa Barbara, University of California, Berkeley), D. Hoelzer (Oak Ridge National Laboratory), and D. Gragg (University of California, Santa Barbara)** **25**

Fully consolidated nanostructured ferritic alloys (NFAs) were prepared by attritor milling pre-alloyed Fe-14Cr-3W-0.4Ti and 0.3wt% Y₂O₃ powders, followed by hot isostatic pressing (HIPing) at 1000°C and 1150°C and 200 MPa for 4 h. Transmission electron microscopy (TEM) revealed similar bimodal distributions of fine and coarse ferrite grains in both cases. However, as expected, the alloy microhardness decreased with increasing in HIPing temperature. Three point bend tests on single edge notched specimens, with a nominal root radius $\rho = 0.15$ mm, were used to measure the notch fracture toughness, K_{Ic} , as a function of test temperature. The K_{Ic} curves were found to be similar for both alloys. It appears that the coarser ferrite grains control cleavage fracture, in a way that is independent of alloy strength and HIPing temperature.

- 3.3 THE MICROSTRUCTURE AND STRENGTH PROPERTIES OF MA957 NANOSTRUCTURED FERRITIC ALLOY JOINTS PRODUCED BY FRICTION STIR AND ELECTRO-SPARK DEPOSITION WELDING—P. Miao, G. R. Odette (University of California, Santa Barbara), J. Gould, J. Bernath (Edison Welding Institute), R. Miller (DDL-OMNI), M. Alinger (University of California, Santa Barbara, University of California, Berkeley) and C. Zanis (DDL-OMNI)** **30**

The nanostructured ferritic alloy (NFA) MA957 was joined by friction stir welding (FSW) and electro-sparked deposition (ESD) welding. Transmission electron microscopy (TEM) and small angle neutron scatter (SANS) characterization studies showed a uniform fine-scale equiaxed ferrite structure with a high density of dislocations and only slightly coarsened nm-scale particles in the joint region of the FSW weld compared to the base metal. Microhardness and tensile measurements on the FSW showed a modest reduction in the strength of the joint compared to the as-processed MA957. In contrast, the ESD welds contained considerable porosity and the nm-scale particles dissolved or coarsened significantly, resulting in a larger degradation of the joint region strength. Thus FSW is a promising method for joining NFAs.

- 3.4 MECHANICAL PROPERTIES OF NEUTRON-IRRADIATED NICKEL-CONTAINING MARTENSITIC STEELS: I. EXPERIMENTAL STUDY—R. L. Klueh, N. Hashimoto, and M. A. Sokolov (Oak Ridge National Laboratory), K. Shiba and S. Jitsukawa (Japan Atomic Energy Research Institute)** **53**

The 9Cr-1MoVNb and 12Cr-1MoVW steels and these steels with 2% Ni additions were irradiated in HFIR to ≈ 10 dpa at 300°C and ≈ 12 dpa at 400°C and in FFTF to ≈ 15 dpa at 393°C. After irradiation in HFIR, steels with 2% Ni hardened more than steels without a nickel addition. When 9Cr-1MoVNb-2Ni, 12Cr-1MoVW, and 12Cr-1MoVW-2Ni steels were irradiated in HFIR at 400°C, they hardened more than when irradiated in FFTF at 393°C. The 9Cr-1MoVNb steel hardened to the same level in both FFTF and HFIR. For all but the 9Cr-1MoVNb steel, shifts in DBTT of the steels irradiated in HFIR were greater than shifts for the same steels irradiated in FFTF. Irradiation in HFIR at 300 and 400°C caused a larger shift for the steels containing 2% Ni than for the steels with no nickel addition. The increase in DBTT was related to the increase in yield stress. Despite the uncertainty inherent in the experiments, the results lead to the conclusion that helium caused an increment of hardening that contributed to the extra shift in DBTT over that observed after irradiation in FFTF where little helium formed.

3.5 ON THE RELATION BETWEEN IRRADIATION INDUCED CHANGES IN THE MASTER CURVE REFERENCE TEMPERATURE SHIFT AND CHANGES IN STRAIN HARDENED FLOW STRESS—G. R. Odette, M. Y. He, and T. Yamamoto (University of California, Santa Barbara)

69

Irradiation hardening produces increases in the cleavage transition fracture toughness reference temperature (ΔT_o). It is traditional to relate ΔT_o to the corresponding changes in the yield stress, $\Delta\sigma_y$, as $C_o = \Delta T_o / \Delta\sigma_y$. However, it is a strain-hardened flow stress, σ_{fl} , in the fracture process zone that controls cleavage, rather than σ_y . Thus, irradiation induced decreases in the strain hardening $\Delta\sigma_{sh}$ (< 0) must be considered along with $\Delta\sigma_y$ (> 0) in evaluating ΔT_o . The $\Delta\sigma_{sh}$ in reactor pressure vessel (RPV) steels irradiated to low doses at around 300°C are small, even for large $\Delta\sigma_y$. However, the $\Delta\sigma_{sh}$ are much greater for high dose irradiations of tempered martensitic steels (TMS) that are candidates for fusion applications. As a result, for the TMS case the C_o are less, and in some instances much less, than for RPV steels and irradiation conditions. We address two key questions. First, how does $\Delta\sigma_{sh}$ influence the $C_o = \Delta T_o / \Delta\sigma_y$ relation? Second, is it possible to derive a universal relation between ΔT_o and $\Delta\sigma_{fl}$ averaged over a pertinent range of ε , $\langle \Delta\sigma_{fl} \rangle$, such that a $C_o' = \Delta T_o / \langle \Delta\sigma_{fl} \rangle$ is independent of the individual values of $\Delta\sigma_y$ and $\Delta\sigma_{sh}$? The results of this study suggest that $\langle \Delta\sigma_{fl} \rangle$ averaged between 0 and 0.1 provides a similar C_o' for various assumptions about the effect of irradiation on $\Delta\sigma_{sh}$. Notably, changes in indentation hardness, ΔH , are also directly related to this same $\langle \Delta\sigma_{fl} \rangle$. Hence, measurements of ΔH should provide a good basis for assessing ΔT_o for a wide range of alloys and irradiation conditions.

3.6 ON THE INTRINSIC INITIATION AND ARREST CLEAVAGE FRACTURE TOUGHNESS OF FERRITE—M. L. Hribernik, G. R. Odette, and M. Y. He (University of California, Santa Barbara)

74

The results of the crack arrest fracture toughness (K_a) measurements on cleavage oriented Fe single crystals from -196 to 0°C are reported. Arrest measurements were performed on four low toughness cleavage orientations; (100)[010], (100)[011], (110)[001] and (110)[110]. Reliable and consistent measurements were obtained for the (100) cleavage planes, however inconsistent measurements were observed for the (110) planes as cleavage cracks always reverted back to the (100) planar orientation. The corresponding static (K_{ic}) and dynamic (K_{id}) cleavage initiation toughness for the (100) planes from -196 to 50°C were also measured over a range of applied stress intensity rates (\dot{K}) from ≈ 0.1 to 10^4 MPa $\sqrt{m/s}$. The $K_{ia}(T)$, $K_{ic}(T)$ and $K_{id}(T)$ gradually increase with temperature from a minimum average K_{ia} value of approximately 4 MPa \sqrt{m} up to a rapid BDT at $\approx 0^\circ\text{C}$. The BDT temperature increases with higher \dot{K} , and is highest for K_{ia} . The \dot{K} dependence of $K_{ic/d}(T)$ is consistent with the strain rate dependence of thermally activated flow stress of Fe. The $K_{ic}(T)$ for single crystal Fe and W are also reasonably similar when plotted on a homologous temperature scale. The $K_{ia}(T)$ for Fe at $\approx -40^\circ\text{C}$ is similar to that for Fe-3wt%Si at $\approx 110^\circ\text{C}$. This 150°C shift can be reasonably rationalized by the solid solution lattice strengthening of Si. The $K_{ia}(T)$ for Fe must be shifted up by $\approx 220^\circ\text{C}$ to approximate the temperature dependence of the $K_{id}(T)$ that is consistent with a universal $K_{ic}(T)$ master curve shape. This magnitude of shift may be caused by a combination of thermally activated (rate-dependent) solid solution lattice strengthening, complemented by long-range internal stress fields.

3.7 SWELLING AND MICROSTRUCTURE OF PURE Fe AND Fe-Cr ALLOYS AFTER NEUTRON IRRADIATION TO ~26 DPA AT 400°C—Yu V. Konobeev, A. M. Dvoriashin, S. I. Porollo (The Institute of Physics and Power Engineering, Russia), F. A. Garner (Pacific Northwest National Laboratory) **80**

The microstructures of pure Fe and various Fe-Cr binary alloys in both the annealed and heavily cold worked conditions were investigated after irradiation to 25.8 dpa at 4×10^{-7} dpa/s in the BR-10 fast reactor. Microscopy has shown that the largest swelling of 4.5% was observed in the cold worked pure iron while that of annealed Fe is only 1.7%. Additions of 2% chromium resulted in a decrease of swelling, but the swelling of cold worked Fe-2Cr alloy was still higher than that of the annealed condition. Independent of the initial starting condition, swelling in the Fe-6Cr alloy was completely suppressed. In alloys with higher chromium content swelling of 0.04–0.05% was observed only in samples irradiated in the annealed condition. There were also significant changes in dislocation and precipitate microstructure following irradiation.

4.0 COPPER ALLOYS **86**

No contributions.

5.0 REFRACTORY METALS AND ALLOYS **87**

No contributions.

6.0 AUSTENITIC STAINLESS STEELS **88**

6.1 THE MECHANISM OF STRESS INFLUENCE ON SWELLING OF 20% COLD WORKED 16Cr15Ni2MoTiMnSi STEEL—I. A. Portnykh, A. V. Kozlov, V. L. Panchenko (FSUE Institute of Nuclear Materials), V. M. Chernov (A. A. Bochvar Institute of Inorganic Materials), and F. A. Garner (Pacific Northwest National Laboratory) **89**

Argon-pressurized tubes of 20% cold-worked 16Cr15Ni2MoTiMnSi steel were irradiated at hoop stresses of 0, 100, and 200 MPa at ~740K in the BN-600 fast reactor to 108 dpa. Following nondestructive measurements of strain, density measurements and microscopy were conducted. Voids were categorized into three types depending on their association with other microstructural features. Stress enhanced the nucleation of all void types, but nucleation of voids associated with dislocations was increased the most. Swelling increased as a consequence, even though the average size of each type void decreases. Swelling measured by TEM and density change gave identical results.

6.2 THE INFLUENCE OF COLD-WORK LEVEL ON THE IRRADIATION CREEP AND SWELLING OF AISI 316 STAINLESS STEEL IRRADIATED AS PRESSURIZED TUBES IN THE EBR-II FAST REACTOR—E. R. Gilbert and F. A. Garner (Pacific Northwest National Laboratory) **95**

Pressurized tubes of AISI 316 stainless steel irradiated in the P-1 experiment in the EBR-II fast reactor have been measured to determine the dependence of irradiation-induced strains resulting from plastic deformation, irradiation creep, void swelling and precipitation. It is shown that the Soderberg relation predicting no axial creep strains in biaxially-loaded tubes is correct for both plastic and creep strains. Swelling strains are shown to be isotropically distributed both for stress-free and stress-affected swelling, while precipitation strains are somewhat anisotropic in their distribution. When corrected for stress-enhancement of swelling, the derived irradiation creep strains appear to be identical for both annealed and 20% cold-worked specimens, and also for tubes strained by rise to power increases in pressure. For relatively small creep strains it is often difficult to separate the creep and non-

creep components of deformation.

- 6.3 RECENT EXPERIMENTAL RESULTS ON NEUTRON-INDUCED VOID SWELLING OF AISI 304 STAINLESS STEEL CONCERNING ITS INTERACTIVE DEPENDENCE ON TEMPERATURE AND DISPLACEMENT RATE—F. A. Garner (Pacific Northwest National Laboratory), B. J. Makenas (Fluor Hanford Company) 104**

The objective of this effort is to explore the response of austenitic steels in diverse nuclear environments. Since light water reactors generate helium/dpa levels comparable to fusion devices, there is considerable overlap in relevance. In addition, the focus on AISI 304, while not of direct application to fusion devices, is useful because this simple steel does not have a very complicated phase evolution, allowing study of radiation-induced microstructural evolution without the complications associated with precipitation.

- 7.0 MHD INSULATORS, COATINGS, INSULATING CERAMICS, AND OPTICAL MATERIALS 115**

- 7.1 INVESTIGATION OF Pb-Li COMPATIBILITY FOR THE DUALCOOLANT TEST BLANKET MODULE—B. A. Pint and J. L. Moser (Oak Ridge National Laboratory) 116**

Static capsule tests in Pb-17Li were performed on coated and uncoated type 316 stainless steel and Al-containing alloys at 800°C and the Pb-Li was analyzed after each capsule test. Chemical vapor deposited (CVD) aluminide coatings on type 316 substrates reduced dissolution by ~50X at 800°C compared with uncoated samples. Little effect of pre-oxidation was observed for the performance of the coating. These results indicate that aluminide coatings may be a viable option to allow conventional Fe- or Ni-base tubing alloys to carry Pb-Li from the first wall to the heat exchanger. Future work will need to include testing in a flowing system with a thermal gradient to fully determine the compatibility of these materials. In order to test the viability of using a thermal convection loop made of quartz, a quartz ampoule was filled with Pb-Li and exposed for 1000h at 800°C. No Si was detected in the Pb-Li after the test indicating that quartz may be a low cost construction option.

- 8.0 BREEDING MATERIALS 120**

No contributions.

- 9.0 RADIATION EFFECTS, MECHANISTIC STUDIES, AND EXPERIMENTAL METHODS 121**

- 9.1 MODELLING THERMODYNAMICS OF ALLOYS FOR FUSION APPLICATION—A. Caro, M. Caro, J. Marian (Lawrence Livermore National Laboratory), E. Lopasso (Centro Atomico Bariloche, Argentina), and D. Crowson (Virginia Polytechnic Institute) 122**

Atomistic simulations of alloys at the empirical level face the challenge of correctly modeling basic thermodynamic properties. In this work we develop a methodology to generalize many-body classic potentials to incorporate complex formation energy curves. Application to Fe-Cr allows us to predict the implications of the ab initio results of formation energy on the phase diagram of this alloy and to get a detailed insight into the processes leading to precipitation of α' phase under irradiation.

- 9.2 THERMAL HELIUM DESORPTION OF HELIUM-IMPLANTED IRON—D. Xu, T. Bus, S. C. Glade, and B. D. Wirth (University of California, Berkeley) 128**

Following the last report, we have performed new implantations at energies lower than 100 keV, with an aim of reducing the penetration depth of helium atoms and the structural damage, and thus reducing the He-point defect interaction complexity during desorption experiments. Initial measurements on the new samples have revealed a large number of

desorption peaks within both the bcc and fcc temperature ranges. These peaks are well fit with first order reaction kinetics, which reveal activation energies ranging from ~1.9 to ~3.5 eV. The number and the relative intensities of detected desorption peaks within the bcc temperature range appear to increase with decreasing implantation energy. Previously reported spurious peaks and a non-1st-order sharp peak are now better understood and described in the current report.

9.3 HELIUM IN IRRADIATED IRON: A MULTI-SCALE STUDY—T. Seletskaya, Yu. N. Osetsky, R. E. Stoller, and G. M. Stocks (Oak Ridge National Laboratory) 140

Helium is produced in neutron-irradiated metals as the result of (n, α) transmutation reactions and plays a significant role in microstructure evolution and mechanical properties degradation [1,2]. Due to helium's high mobility via an interstitial migration mechanism and its strong binding with vacancies, information on its atomistic behavior is hard to assess from experiments. The only way to obtain such information is from first-principles electronic structure calculations. However, electronic structure calculations can not be used on the time and size scales needed to simulate the important evolution of helium-vacancy clusters that ultimately leads to bubble formation. A multi-scale approach, based on constructing an empirical potential and employing this potential in classical molecular dynamics, seems to be the only practical approach currently available to study He behavior in metals on the desired scale.

9.4 MODELING THE INTERACTION OF HELIUM WITH DISLOCATIONS AND GRAIN BOUNDARIES IN ALPHA-IRON—H. L. Heinisch, F. Gao, and R. J. Kurtz (Pacific Northwest National Laboratory) 147

Molecular statics, molecular dynamics and the dimer method of potential surface mapping are being used to study the fate of helium in the vicinity of dislocations and grain boundaries in alpha-iron. Even at very low temperatures interstitial helium atoms can migrate to dislocations and grain boundaries, where they are strongly bound. The binding energies of helium to these microstructural features, relative to the perfect crystal, and the migration energies of helium diffusing within them have a strong correlation to the excess atomic volume that exists in these extended defects. Helium atom migration energies within the dislocations and grain boundaries studied are in the range of 0.4–0.5 eV. Helium “kick out” mechanisms have been identified within dislocations and grain boundaries by which interstitial helium atoms replace a Fe lattice atom, creating a stable He-vacancy complex that may be a nucleation site for a He bubble.

10.0 DOSIMETRY, DAMAGE PARAMETERS, AND ACTIVATION CALCULATIONS 154

No contributions.

11.0 MATERIALS ENGINEERING AND DESIGN REQUIREMENTS 155

No contributions.

12.0 IRRADIATION FACILITIES AND TEST MATRICES 156

12.1 IRRADIATION OF FUSION MATERIALS IN THE BR2 REACTOR: THE FRISCO-F EXPERIMENT—E. Lucon (SCK•CEN) and M. A. Sokolov (Oak Ridge National Laboratory) 157

Tensile and miniature Compact Tension specimens of eight high chromium steels of fusion relevance have been irradiated in the BR2 reactor in the framework of a collaborative project between Oak Ridge National Laboratory (ORNL) and SCK•CEN. All samples have been irradiated at a nominal temperature of 300°C in the in-pile section 2 (IPS-2) for five cycles (02/2005 to 01/2006) up to an average fast neutron fluence of 8.02

× 1020 n/cm² or 1.20 dpa. The rig was rotated three times by 180° in order to reduce the radial and azimuthal neutron flux gradients.