

**DOE-ER-0313/37
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Categories
UC-423, -424**

**FUSION MATERIALS
SEMIANNUAL PROGRESS REPORT
FOR THE PERIOD ENDING
December 31, 2004**

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

DATE PUBLISHED: MARCH 2005

**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 thirty-seventh 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

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Fast fracture properties of chemically vapor-infiltrated silicon carbide matrix composites

with Hi-Nicalon™ Type-S near-stoichiometric silicon carbide fiber reinforcements and thin pyrolytic carbon interphase were studied. The primary emphasis was on preliminary assessment of the applicability of a very thin pyrolytic carbon interphase between fibers and matrices of silicon carbide composites for use in nuclear environments. It appears that the mechanical properties of the present composite system are not subject to strong interphase thickness effects, in contrast to those in conventional non-stoichiometric silicon carbide-based fiber composites. The interphase thickness effects are discussed from the viewpoints of residual thermal stress, fiber damage, and interfacial friction. A preliminary conclusion is that a thin pyrolytic carbon interphase is beneficial for fast fracture properties of stoichiometric silicon carbide composites.

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3.1 REDUCED-ACTIVATION BAINITIC AND MARTENSITIC STEELS FOR NUCLEAR FUSION APPLICATIONS—R. L. Klueh (Oak Ridge National Laboratory) 40

Reduced-activation steels were developed to enhance safety and reduce adverse environmental effects of future fusion power plants. Martensitic and bainitic steels were developed during the 1985–90 timeframe, and the feasibility of their use for fusion was investigated in an international collaboration from 1994 to present. Work continues to improve the steels and understand the effect of neutron irradiation on them.

3.2 FRICTION STIR WELDING OF MA957 OXIDE DISPERSION STRENGTHENED FERRITIC STEEL—S. M. Howard, B. K. Jasthi, and W. J. Arbegast (South Dakota School of Mines and Technology), G. J. Grant, S. Koduri, D. R. Herling, and D. S. Gelles (Pacific Northwest National Laboratory) 55

A 1-in. (25.4 mm) diameter yttria-dispersion-strengthened MA957 ferritic steel alloy tube with a 0.125" (3.18 mm) wall thickness was successfully plasticized by friction stir welding. The pin tool was a W-Re tool with 0.125" (3.17 mm) diameter tip. It showed no discernable wear for the total 12" (305 mm) of weld. Weld conditions were 1000 and 1400 RPM, 4 in/min (101 mm/min), with and without preheating to 135°C. Metallographic analysis of the post friction-stir welded material showed a decrease in material hardness to 225±22 HV compared to the parent material at 373±21 HV. All weld conditions produced plasticization; however, improved plasticization was observed for preheated samples.

3.3 ON THE PRECIPITATION KINETICS, THERMAL STABILITY AND STRENGTHENING MECHANISMS OF NANOMETER SCALE Y-Ti-O CLUSTERS IN NANOSTRUCTURED FERRITIC ALLOYS—M. J. Alinger and G. R. Odette (University of California, Santa Barbara) 61

A systematic matrix of annealing times and temperatures were used to assess the kinetics of NC precipitation in Fe-14Cr powders mechanically alloyed (MA) with Ti and Y2O3 (U14YWT). The MA dissolves the Y, O, and Ti as supersaturated solutes that subsequently precipitate during hot powder consolidation, or annealing, in the form of nm-scale solute clusters (NCs). The NCs evolve extremely rapidly due to high diffusion rates and excess vacancies produced by MA. The non-equilibrium kinetics of NC evolution is nucleation controlled, with the number density (N) scaling with an effective activation energy of $\approx 53 \pm 15$ kJ/mole. The stability of the NCs during high-temperature annealing of MA957 was also characterized. The NCs coarsen and transform to nearer-to-equilibrium oxide phases at radii $\gg 3.5$ nm, with a high effective activation energy of ≈ 880 kJ/mole and a time dependence characteristic of a dislocation pipe diffusion mechanism, with $r(t) - r(0) \propto t^{1/5}$. The effect of the micro-nanostructure on the alloy strength was assessed by microhardness measurements. The NCs can be sheared by

dislocations and have an obstacle strength (α) that increases with r (nm) as $\alpha \approx 0.37 \log(r/2b)$ (≈ 0.1 to 0.5).

- 3.4 POSITRON ANNIHILATION CHARACTERIZATION OF NANOSTRUCTURED FERRITIC ALLOYS**—M. J. Alinger and G. R. Odette (University of California, Santa Barbara), S. C. Glade and B. D. Wirth (University of California, Berkeley), Y. Nagai and M. Hasegawa (Tohoku University, Sendai Japan) **68**

Positron annihilation spectroscopy lifetime and orbital electron momentum spectroscopy (OEMS) data are in qualitative agreement with small angle neutron scattering (SANS) observations for NCs in NFAs. In the U14WT alloys, which do not contain yttrium, the positrons primarily annihilate in the matrix and matrix features like dislocations or small solute clusters. A small fraction of the positrons annihilate at large vacancy clusters or gas bubbles. In the case of the Y containing alloy, U14YWT, up to $\approx 50\%$ of the positrons annihilate at non-magnetic features characteristic of Y-Ti-O NCs and, perhaps, smaller vacancy cluster-bubble type features.

- 3.5 A STATISTICAL APPROACH TO FRACTURE TOUGHNESS MODELING OF MA957 USING A σ^* - A^* CONCEPT**—W. J. Yang, M. J. Alinger, T. Yamamoto, and G. R. Odette (University of California, Santa Barbara) **82**

We modeled the temperature (T) dependent fracture toughness $KJc(T)$ of MA957 based on a statistically modified critical stress-critical stressed area (σ^* - A^*) concept. The finite element (FE) method was used to simulate the stress-strain fields as a function of the applied loading KJ at different T in terms of the area (A) encompassed by a specified normal stress contour (σ). Ideally the critical stress (σ^*) is defined by the point of intersection of $A(\sigma)$ plots at various T . However, a statistically mediated range of A^* was recognized in our model, corresponding to the intrinsic distribution of KJc : thus the point at which the $A(\sigma)$ at various T experience the maximum number of intersections was used to define σ^* . The fracture toughness of MA957 is strongly dependent on the specimen orientation. Analysis of cleavage initiation in the L-R orientation, with the highest KJc yielded the highest $\sigma^* \approx 3600$ MPa. In contrast, the σ^* for the C-L orientation, with the lowest KJc , yielded the lowest $\sigma^* \approx 2850$ MPa, while for the C-R orientation with intermediate KJc , $\sigma^* \approx 3000$ MPa. In the latter two cases, the ligament planes contain directions parallel to the extrusion direction. However, the A^* were roughly similar for all orientations ranging from ≈ 30 to $400 \mu m^2$. This is probably a result of a common distribution of cleavage initiation sites in the form of μm -scale Al_2O_3 particles aligned in the extrusion direction. The A^* - σ^* , was used to model median $KJc(T)$ and the corresponding curves at high and low fracture probabilities determined from a Weibull analysis. The model is in good agreement with previously measured KJc data, but requires a K_{min} of $10 \text{ MPa}\sqrt{m}$ in the C-L orientation, which less than the standard Master Curve (MC) value of $20 \text{ MPa}\sqrt{m}$. We conclude that the low toughness direction is due to both intrinsic crystallographic and microstructurally mediated factors.

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No contributions

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- 5.1 MICROSTRUCTURAL ANALYSIS ON HELIUM RETENTION OF ION-IRRADIATED AND ANNEALED TUNGSTEN FOILS**—N. Hashimoto and J. D. Hunn (Oak Ridge National Laboratory), N. Parikh, S. Gilliam, S. Gidcumb, and B. Patnaik (University of North Carolina at Chapel Hill), and L. L. Snead (Oak Ridge National Laboratory) **91**
- The helium retention characteristics and helium bubble distribution in tungsten were

studied using $^3\text{He}(d,p)^4\text{He}$ nuclear reaction analysis (NRA) and transmission electron microscopy (TEM) on two forms of tungsten: single crystal and polycrystalline, implanted to 1×10^{19} $^3\text{He}/\text{m}^2$ at 850°C and annealed at 2000°C . The NRA results revealed that as-implanted single crystal and polycrystalline tungsten exhibited similar helium retention characteristics. Stepwise annealing reduced the helium retention in both single crystal and polycrystalline tungsten when the number of implantation steps and annealing time were increased. The TEM results indicated that microstructure played a large role in helium trapping; the existence of grain boundaries led to significant cavity formation and greater cavity growth. Single crystal tungsten had less trapping sites for helium, allowing long range He diffusion during annealing. The decrease of He retention in polycrystalline tungsten during stepwise annealing was probably due to significant recrystallization, resulting in decrease of grain boundary density.

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A new and powerful indentation hardness (H) approach to evaluating the true stress (σ)-true plastic strain (ϵ), $\sigma(\epsilon)$, constitutive behavior of materials is described. Since measurements of H intrinsically probe a wide-range of ϵ (up to ≈ 0.5), accurate assessment of the corresponding yield (σ_y) stress and strain hardening [$\sigma_{sh}(\epsilon)$] pose a significant challenge. Extensive elastic-plastic finite element (FE) simulations have been carried out to assess the relation between H and $\sigma(\epsilon)$. The simulations were based on both a wide variety of analytical $\sigma(\epsilon)$ relations, in the form of $\sigma(\epsilon) = \sigma_y + \sigma_{sh}(\epsilon)$, as well as actual $\sigma(\epsilon)$ derived from data on a large number of alloys with a very wide range of constitutive behavior. The analysis led to derivation of a remarkable universal relation between H and $\sigma(\epsilon)$ given by $H \approx 4.05(1 + 34.6\sigma_{flow}/E)\sigma_{flow}$, where $\sigma_{flow} = \sigma_y + \langle\sigma_{sh}\rangle$, $\langle\sigma_{sh}\rangle$ is the average strain hardening between $\epsilon = 0$ and 10%, and E is the elastic modulus. Note we use consistent MKS units of MPa for both H and σ_{flow} . The expression for $H(\sigma_{flow})$ also can be inverted to one describing $\sigma_{flow}(H)$. Experimental σ_{flow} -H data

pairs based on this definition of σ_{flow} for the large set of alloys noted above with a very diverse range of $\sigma(\epsilon)$ are in excellent agreement with the model predictions. The σ_{flow} - H relation provides insight into the large variation of the H/σ_y ratios that are observed for different materials, as well as the corresponding variation in the $\Delta H/\Delta\sigma_y$ ratios used to estimate $\Delta\sigma_y$ due to irradiation based on measurements of ΔH . Applications of the H/σ_{flow} relation, including both evaluating $\langle\sigma_{sh}\rangle$ in materials that have very low uniform strain capacity in standard tensile tests and measuring at $\sigma(\epsilon)$ high ϵ .

9.2 THE INTERACTION OF HELIUM ATOMS WITH EDGE DISLOCATIONS IN α -Fe—H. L. Heinisch, F. Gao, R. J. Kurtz, and E. A. Le (Pacific Northwest National Laboratory) **116**

Formation energies, binding energies, and migration energies of interstitial He atoms in and near the core of an $a/2\langle 111 \rangle\{110\}$ edge dislocation in α -Fe are determined in atomistic simulations using conjugate gradient relaxation and the Dimer method for determining saddle point energies. Results are compared as a function of the proximity of the He to the dislocation core and the excess interstitial volume in regions around the dislocation. Interstitial He atoms have negative binding energy on the compression side of the dislocation and strong positive binding energy on the tension side. Even at low temperatures, interstitial He atoms in the vicinity of the dislocation easily migrate to the dislocation core, where they form crowdion interstitials oriented along the close-packed slip direction, with binding energies in excess of 2 eV. Crowdion interstitial He atoms diffuse along the core, transverse to the crowdion direction, with a migration energy of 0.4–0.5 eV.

9.3 MOLECULAR DYNAMICS SIMULATION OF PRIMARY IRRADIATION DEFECT FORMATION IN FE-CR ALLOYS—J.-H. Shim, H.-J. Lee, and B. D. Wirth (University of California, Berkeley) **120**

Molecular dynamics simulations of displacement cascades up to 20 keV have been performed in Fe and Fe-10%Cr using two different parameterizations of Finnis-Sinclair type interatomic potentials. The two different potentials describe the extremes of positive (attractive) and negative (repulsive) binding between substitutional Cr atoms and Fe self-interstitial atoms. The effect of Cr, regardless of potential, has a minimal effect on the collisional stage of cascade and on the distribution and number of vacancy and self-interstitial atom clusters. The quantity of mixed Fe-Cr dumbbells is sensitive to the choice of potential, however.

9.4 MODELLING THERMODYNAMICS OF ALLOYS FOR FUSION APPLICATION—A. Caro (Lawrence Livermore National Laboratory) (Work done in collaboration with B. Sadigh, P. E. A. Turchi, and M. Caro, Lawrence Livermore National Laboratory) **133**

The real problem of microstructure evolution of steels under irradiation involves complex thermodynamics of non-equilibrium and kinetic processes in multicomponent–multiphase alloys. Empirical simulations have a long history addressing Fe and, in particular, with one impurity relevant in fission reactor pressure vessels steels: Cu. However no realistic assessment of the accuracy of the model predictions has been available until very recently. The classic computational approach deals with an oversimplified description of alloys.

9.5 HELIUM BEHAVIOR IN METALS CHARACTERIZED BY THERMAL HELIUM DESORPTION SPECTROSCOPY—S. C. Glade and B. D. Wirth (University of California, Berkeley) and H. Schut (Interfaculty Reactor Institute, Delft University of Technology, Delft, The Netherlands) **136**

A facility to perform thermal helium desorption spectroscopy is under construction at the

University of California, Berkeley. The facility is scheduled to be validated and in operation in early June 2005. Experiments are currently being designed to provide data on helium diffusion, trapping mechanisms and energetics in iron and iron alloys.

9.6 HELIUM – SELF-INTERSTITIAL ATOM INTERACTION IN FERRITIC ALLOY—L. Ventelon and B. D. Wirth (University of California, Berkeley), C. Domain (Electricite de France) 140

Atomistic simulations have been performed to investigate the effect of He impurities on the properties and behavior of self-interstitial atom clusters in Fe. Ferritic alloys are candidate fusion energy first wall and breeding blanket structural materials, and will be exposed to high levels of radiation damage and transmutation products in a 14 MeV peaked fusion neutron spectrum. A comparison is made of the interaction energies between interstitial He atoms and a single self-interstitial atom (SIA) obtained with ab-initio electronic structure and semi-empirical interatomic potentials using molecular dynamics and conjugate gradient molecular statics calculations. The results provide insight into the validity of semi-empirical interatomic potentials and a basis for extrapolating ab-initio results from small to larger system sizes. We also present the results of MD investigation into the migration behavior of SIAs and SIA clusters in the presence of interstitial and substitutional He. The MD simulations reveal a strong interaction between He and SIA clusters, often resulting in SIA – vacancy reactions that spontaneously eject helium into interstitial sites, and provide quantitative information on the interaction radii, trapping – binding energetics and migration behavior of mixed He-SIA clusters.

9.7 DIFFUSION OF He INTERSTITIALS IN GRAIN BOUNDARIES IN α -Fe—F. Gao, H. L. Heinisch, and R. J. Kurtz (Pacific Northwest National Laboratory) 151

The migration and diffusion mechanisms of interstitial He atoms at two representative grain boundaries in α -Fe, $\Sigma 11\langle 110 \rangle \{323\}$ and $\Sigma 3\langle 110 \rangle \{112\}$, are studied using molecular dynamics. The migration of He atoms was followed for 10 – 30 ns, at temperatures between 600 and 1200 K. The diffusion coefficient of He atoms is calculated using the mean square displacements of He atoms, and the effective migration energies were determined to be 0.32 and 0.44 eV for $\Sigma 11\langle 110 \rangle \{323\}$ and $\Sigma 3\langle 110 \rangle \{112\}$ GBs, respectively. He interstitial diffuse quickly in the $\Sigma 11$ GB with one-dimensional behavior along the GB directions, while it migrates one-dimensionally at low temperature, two-dimensionally at intermediate temperature and three-dimensionally at higher temperature in the $\Sigma 3$ GB. The different activation energies and diffusion mechanisms in these two representative grain boundaries suggests that the varying atomic structures of the grain boundaries are important for the diffusivity of He.

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The 17J experiment has been irradiated for 3 of 10 planned cycles in the HFIR. The upper capsule has been operating 20 to 50°C below the target temperature of 700°C for

the last two cycles. The cause has not been determined. The middle capsule has operated at its target temperature of 600°C. A partial blockage of the purge gas line observed in the first cycle of irradiation led to a reduction in the target temperature for the lower capsule to ~425°C (from an original target of 450°C). Further degradation of this blockage has caused some operational difficulties resulting in this capsule running at slightly lower temperatures, ~418°C average, for part of cycle 402.