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**FUSION MATERIALS
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FOREWORD

This is the thirty-sixth 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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	Near-stoichiometric SiC fiber composites with polymer-derived high-crystallinity SiC matrices were produced and were characterized their microstructures and mechanical properties. The material was produced through repeated polymer-impregnation and pyrolysis (PIP) cycles of Tyranno™-SA sintered SiC fibers, following pyrolytic carbon and SiC bi-layer interphase deposition. Co-polymer of polycarbosilane (PCS) and polymethylsilane (PMS) was used as the precursor for near-stoichiometric SiC matrix. The final heat treatment was performed at 1100 – 1800°C in flowing commercial-purity argon. The produced composites were dense in general with small macro-porosity. Transmission electron microscopy revealed that the matrix microstructures after heat treatment at $>1500^\circ\text{C}$ consisted of fine layers of SiC crystallites in amorphous SiC and carbon matrix. High temperature heat treatment promoted crystallization at the expense of porosity. No degradation of Tyranno-SA fiber was identified after heat treatment at 1800°C. PIP-composites with well-crystallized SiC matrices exhibited flexural fracture behavior very similar to that of chemically vapor infiltrated composites. The composites maintained ambient temperature strength up to 1000°C in air and to 1300°C in argon.	

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A new approach to joining SiC-based ceramics is described and evaluated for Fusion Energy systems. The joining method is based on pre-ceramic polymers filled with reactive and inert filler powders and is similar to other approaches that use such materials. This approach differs in the particular polymer system and in the details of the processing. A principal advantage of this approach relative to other, similar approaches is that the polymer system is easily handled in ambient air and can be processed in air. This makes the joining process simple and field repairable. The joining compound is a liquid that can be painted, sprayed, or applied by dip coating.

2.3 STRENGTH AND ELASTIC MODULUS OF NEUTRON-IRRADIATED CUBIC SILICON CARBIDE—Y. Katoh and L. L. Snead (Oak Ridge National Laboratory) **33**
 "The corrected version of this contribution was posted February 23, 2006"

Mechanical properties of high purity polycrystalline cubic silicon carbide were characterized after neutron irradiation. The materials were irradiated in target position capsules in High Flux Isotope Reactor to nominal neutron fluence levels of up to 7.7 dpa at temperatures of 300, 500 and 800°C. Reduction in Young's modulus was observed after irradiation, and its irradiation temperature dependence agreed qualitatively with calculated modulus change due to point defect swelling. Irradiation caused very significant modification of statistical flexural strength but caused only minor increase in nano-indentation hardness. It was pointed out that the irradiation effect on fracture initiation through an enhanced cleavage resistance in large grains could be contributing to the major change in flexural strength properties.

2.4 MODELING THE TRANSVERSE THERMAL CONDUCTIVITY OF 3D-SiC_f/SiC COMPOSITES—G. E. Youngblood and R. H. Jones (Pacific Northwest National Laboratory) and Reiji Yamada (JAERI, Tokai-mura, JP) **44**

Our previously developed hierarchical two-layer (H2L) model was modified to describe the effective transverse thermal conductivity (K_{eff}) of a three-dimensional (3D) SiC/SiC composite plate made with cross-layered and Z-stitched X:Y:Z uniaxial fiber tow sub-units. As before, the model describes K_{eff} in terms of constituent, microstructural and architectural properties that include the expected effects of fiber-matrix interfacial conductance, of high fiber packing fractions within individual tow sub-units and of the non-uniform porosity contents, shapes and orientations within these sub-units. Model predictions were obtained for two versions of a 3D-Tyranno SA™/PyC/ICVI-SiC composite that had similar fiber/matrix pyrocarbon (PyC) interfaces, relatively high bulk densities (~2.88 g/cc), and an X:Y configuration with fiber content ratios 1:1. The only major difference between the two versions was their Z-stitch fiber content where the relative fiber ratios were 0.1 and 1.2 in the Z sub-units.

2.5 FAST FRACTURE STRENGTH OF THIN PYROLYTIC CARBON INTERPHASE SILICON CARBIDE COMPOSITES—Y. Katoh, T. Nozawa, and L. L. Snead (Oak Ridge National Laboratory), T. Hinoki and A. Kohyama (Kyoto University, Japan), and W. Yang (National Institute for Materials Science, Japan) **52**

The applicability of very thin pyrolytic carbon (PyC) interphase between fibers and matrices in silicon carbide (SiC) fiber-reinforced, chemically vapor-infiltrated SiC matrix composites was studied based on investigations on the effect of interphase thickness on fast fracture properties. It appears that the mechanical properties of near-stoichiometric high-crystallinity SiC fiber composites are not subject to strong interphase thickness

effect, which has been reported for non-stoichiometric SiC fiber composites. This difference was discussed from the viewpoints of thermal residual stress, process-induced damages, fiber surface features, and interfacial bonding and friction. A preliminary conclusion is drawn that a thin PyC interphase, as low as ~25nm, will be generally beneficial for both fast fracture and lifetime limiting properties in stoichiometric SiC-based composites.

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Oxide dispersion-strengthened (ODS) steels are being developed and investigated for nuclear fission and nuclear fusion applications in Japan, Europe, and the United States. In addition, commercial ODS products are available and have been used in niche applications. Microstructural and mechanical properties studies have been conducted at Oak Ridge National Laboratory and elsewhere on various commercial and experimental ODS steels. Tensile and creep properties have been obtained and collected from literature and commercial sources. These data are compared to show the differences and similarities of different ODS steels, and observations are explained in terms of the microstructures of the steels.

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No contributions

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Static lithium compatibility tests have been conducted on vanadium alloys at 800°C. Mass gains have been detected that are not consistent with prior static Li exposures of unalloyed vanadium and are difficult to interpret based on the impurity levels in the lithium specified by the manufacturer. A second test gave more reasonable results. Some of the mass gain could be attributed to N and C uptake from the Li but this should be countered by O loss from the alloy. Post exposure analysis of the specimens indicated a surface layer rich in O, C and Ca but no indication for the embrittlement observed.

- 7.2 EVALUATION OF THE CHEMICAL COMPATIBILITY OF Y_2O_3 AS AN ELECTRICALLY INSULATING COATING FOR A LIQUID LITHIUM BLANKET**—P. F. Tortorelli, B. A. Pint, and T. M. Besmann (Oak Ridge National Laboratory, USA) **81**

Modeling of the chemical stability of Y_2O_3 in molten lithium was hampered by the lack of available thermochemical data for mixed oxides of Li-Y and Ti-Y. Nevertheless, this evaluation indicated that Y_2O_3 was not stable in a lithium environment at 600-1000°C. Furthermore, the results indicated that the experimental observations of Y-Ti oxides on Y_2O_3 after exposure to molten lithium in V-4Ti-4Cr capsules was likely due to formation of Li_2TiO_3 and $LiYO_2$.

- 7.3 HIGH DOSE NEUTRON IRRADIATION OF $MgAl_2O_4$ SPINEL: EFFECTS OF POST-IRRADIATION THERMAL ANNEALING ON EPR AND OPTICAL ABSORPTION**—A. Ibarra (CIEMAT. Inst. Investigación Básica), D. Bravo (Universidad Autonoma de Madrid), F. J. Lopez (Universidad Autonoma de Madrid), and F. A. Garner (Pacific Northwest National Laboratory) **84**

Electron paramagnetic resonance (EPR) and optical absorption spectra were measured during thermal annealing for stoichiometric $MgAl_2O_4$ spinel that was previously irradiated in FFTF-MOTA at ~405°C to ~50 dpa. Both F and F+ centers are to persist up to very high temperatures (over 700°C), suggesting the operation of an annealing mechanism based on evaporation from extended defects. Using X-ray irradiation following the different annealing steps it was shown that the optical absorption band is related to a sharp EPR band at $g=2.0005$ and that the defect causing these effects is the F+ center.

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- 9.1 MULTISCALE MODELING OF DEFECT GENERATION AND EVOLUTION IN METALLIC ALLOYS FOR FUSION POWER**—Jörg Rottler, David J. Srolovitz, and Roberto Car (Princeton Institute for the Science and Technology of Materials, Princeton University) **94**

We perform an analysis of the time evolution of self-interstitial atom and vacancy (point defect) populations in pure bcc metals under constant irradiation flux conditions. Mean-field rate equations are developed in parallel to a kinetic Monte Carlo (kMC) model. When only considering the elementary processes of defect production, defect migration, recombination and absorption at sinks, the kMC model and rate equations are shown to be equivalent and the time evolution of the point defect populations is analyzed using simple scaling arguments. We show that the typically large mismatch of the rates of interstitial and vacancy migration in bcc metals can lead to a vacancy population that grows as the square root of time. The vacancy cluster size distribution under both irreversible and reversible attachment can be described by a simple exponential function. We also consider the effect of highly mobile interstitial clusters and apply the model with parameters appropriate for vanadium and iron.

- 9.2 IN-SITU TEM STUDY ON THE COLLAPSE PROCESS OF SFT DURING PLASTIC DEFORMATION**—Y. Matsukawa, Yu. N. Osetsky, R. E. Stoller, and S. J. Zinkle (Oak Ridge National Laboratory) **98**

Dynamic observation of the collapse process of SFTs during plastic deformation in quenched gold was carried out using a TEM. SFTs having perfect pyramid structure were

collapsed by direct interaction with gliding screw dislocations. Although Wirth et al. pointed out that truncation of SFT before interaction with gliding dislocations was an important factor for the SFT collapse [1], the present experimental results clearly showed that truncation of SFT was not a crucial factor for the collapse mechanism. An SFT collapsed in a unique way: only the base portion divided by the gliding plane of dislocation annihilated, while the apex portion remained intact. Judging from the fact that similar collapse process was observed in recent computer simulations using molecular dynamics codes [2], this collapse process is a major one. However, it is still unclear whether this is only one process for SFT collapse, because this collapse process leaves a small SFT as a remnant, whereas SFTs are completely swept out in the dislocation channels actually observed in the deformation microstructure of irradiated fcc metals [3-12]. Further research works will be required for fully understanding of dislocation channeling, which is believed to be a key for ductility reduction of irradiated metals [3].

9.3 VOID HARDENING IN BCC-IRON STUDIED BY ATOMIC-SCALE MODELLING—S. I. Golubov, Yu. N. Osetsky, and R. E. Stoller (Oak Ridge National Laboratory) 108

Atomic-scale modelling permits detailed simulation of the interactions between moving dislocations and particular obstacles. Such simulations should be of large enough scale to simulate a realistic dislocation density and obstacle spacing, and correctly treat long-range self-interaction between dislocation segments. Results obtained with a 2 nm void and two different atomic-scale techniques based on (1) a periodic array of dislocations (PAD) and (2) Green's function boundary conditions (GF) in α -Fe are presented. Static, zero temperature, simulations have been carried out with incrementally increasing strain until dislocation overcomes the obstacle. It is concluded that both techniques reproduce the same critical resolved shear stress (CRSS), and similar void and dislocation modifications are observed.

9.4 THE INTERACTION OF HELIUM ATOMS WITH EDGE DISLOCATIONS IN α -Fe—H. L. Heinisch, F. Gao, and R. J. Kurtz (Pacific Northwest National Laboratory) 112

Formation energies, binding energies, and the migration of interstitial He atoms at and near the center of an $a/2\langle 111 \rangle\{110\}$ edge dislocation in α -Fe are determined using molecular dynamics and conjugate gradient relaxation methods. Results are compared as a function of the distance of the interstitial He atoms from the center of the dislocation and the amount of excess volume 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 positions near the center of the dislocation, where they form crowdion interstitials with binding energies in excess of 2 eV.

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No contributions

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- The 17J experiment was installed in a europium-shielded RB* position of HFIR and irradiation began with cycle 400 on April 27, 2004. The capsule temperatures were maintained near the target values with the exception of the lower capsule. An apparent temperature-dependent blockage of the purge gas line limited operation of the lower capsule to ~ 425°C.
- 12.2 ASSEMBLY OF THE US-JAPAN JP-27 EXPERIMENT AND START OF IRRADIATION IN THE HFIR—D. K. Felde, K. R. Thoms, D. W. Heatherly, S. H. Kim, R. G. Sitterson, and R. E. Stoller (Oak Ridge National Laboratory) and H. Tanigawa (Japan Atomic Energy Research Institute, Tokai, Japan) 124**
- Specimen and capsule parts fabrication for JP-27 was completed. Loading of specimens into specimen holders and assembly of the capsule was completed. The experiment was installed in the target region of HFIR and irradiation began with cycle 400, starting April 27, 2004.