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A brief assessment of the results of irradiation tests of vanadium alloys conducted in the HFIR-11J, -12J, 13J, and 10J, indicate that significant uncertainties in the data exist which affect the reliability of the data. This assessment concludes that vanadium specimens from the 12J experiment were severely oxidized, and that the irradiation data are unreliable. There is a significant uncertainty in the irradiation temperature for specimens in the 11J, 12J and 13J experiment due partially to gap conductance in the experiment. The very high irradiation creep rates obtained for the vanadium alloys in the 12J experiment are attributed to a large thermal creep component. A detailed thermal analysis of each specimen is required to provide a reliable temperature specification for the data obtained in these experiments. Similar temperature uncertainties may also exist for other materials irradiated in similar test assemblies.

**1.6 CORRECTIONS AND CLARIFICATIONS ON THE EVALUATION OF THE DHCE EXPERIMENT** - D. L. Smith (Argonne National Laboratory) 28

A critical issue in the development of structural materials for the fusion application involves the effects of high helium generation rates on the performance limits of neutron-irradiated materials. Since we do not have a high flux neutron source with fusion-relevant energies, we must rely on simulation techniques to obtain experimental information on these effects. The Dynamic Helium Charging Experiment (DHCE) provides a unique approach for simulating the helium production rates in vanadium alloys in fission reactor irradiations. An assessment of the DHCE-1 proof-of-principle experiment and a subsequent evaluation of the DHCE concept have been presented in the last two semiannual reports. This report attempt to correct and clarify several misinterpretations, incorrect statements and misleading conclusions from the evaluation, which contributed to the decision not to conduct a second DHCE experiment in the early phases of the JUPITER-II collaboration. Specific responses to statements and conclusions presented in the evaluation are presented in this report.

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**2.1 SPECIMEN SIZE EFFECT ON THE IN-PLANE SHEAR PROPERTIES OF SILICON CARBIDE/SILICON CARBIDE COMPOSITES** - T. Nozawa<sup>1</sup>, E. Lara-Curzio<sup>2</sup>, Y. Katoh<sup>1,3</sup>, L.L. Snead<sup>2</sup> and A. Kohyama<sup>1,3</sup> 35

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Miniaturization of test specimens is often necessary to evaluate the physical and mechanical properties of materials under severe environments. The validation of these techniques requires an understanding of the role of geometric and volumetric (size) effects on the mechanical behavior of the material. Although considerable work has been dedicated to understand size and geometric effects on the off-axis tensile strength of continuous fiber-reinforced ceramic matrix composites, little work has been focused on the effect these variables on their shear properties. This paper will present the results of a study aimed at assessing the effect of notch separation and specimen thickness on the shear strength of a 2-D SiC/SiC composite by the Iosipescu test method. Provisions for mounting miniature test specimens using a fixture for standard size specimens are discussed.

## **2.2 TENSILE PROPERTIES OF STOICHIOMETRIC SILICON CARBIDE FIBER REINFORCED FCVI DERIVED SILICON CARBIDE MATRIX COMPOSITES -** 40

T. Nozawa<sup>1</sup>, K. Hironaka<sup>1</sup>, T. Taguchi<sup>2</sup>, N. Igawa<sup>2</sup>, L.L. Snead<sup>3</sup>, Y. Katoh<sup>1</sup>, S. Jitsukawa<sup>2</sup> and A. Kohyama<sup>1</sup>-

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Recently developed SiC/SiC composites with high-crystalline, near stoichiometric SiC fiber are one of the promising materials for fusion and other high-temperature materials, because of the excellent physical and mechanical stability at high-temperature. Therefore material development has been enthusiastically carried out at ORNL as a part of US-Japan collaboration. The objective of this study is to clarify good performance of these composites at severe environment and also to identify the key issues for material development; effects of the interphase thickness, fabric orientation, and porosity on tensile properties, by using small specimen tensile test technique. It was shown that the maximum stress of Tyranno™-SA/FCVI-SiC composites was stable under high-temperature exposure up to 1300°C in mild oxidizing environment. In addition, it was revealed that Tyranno™-SA/FCVI-SiC with single PyC interphase had its maximum strength, when the thickness of PyC was around 150~200 nm.

## **2.3 OPTIMIZING THE FABRICATION PROCESS FOR SUPERIOR MECHANICAL PROPERTIES IN THE STOICHIOMETRIC SiC FIBER REINFORCED FCVI SiC MATRIX COMPOSITE SYSTEM -** 47

T. Taguchi<sup>a</sup>, N. Igawa<sup>a</sup>, T. Nozawa<sup>b</sup>, K. Hironaka<sup>b</sup>, L. L. Snead<sup>c</sup>, T. Hinoki<sup>c</sup>, Y. Katoh<sup>b</sup>, S. Jitsukawa<sup>a</sup>, A. Kohyama<sup>b</sup> and J. C. McLaughlin<sup>c</sup>

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Optimization of the fabrication for SiC composites with stoichiometric SiC fibers (Hi-Nicalon Type S and Tyranno SA) by the forced thermal-gradient chemical vapor infiltration (FCVI) process was carried out. Density and mechanical properties were improved by increasing the fiber volume fraction and optimizing precursor gas flow rates. Porosity was decreased to approximately 15%. Uniformity of fiber/matrix interphase was improved by changing the upstream side and downstream side of a preform during deposition. The tensile strength was seen to slightly increase with thickness of carbon interphase in the range of 75-300 nm. From these results, a dense 300 mm diameter SiC/SiC composite

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**2.4 MODELING THE TRANSVERSE THERMAL CONDUCTIVITY OF 2D-SiC/SiC COMPOSITES** – G. E. Youngblood, D. J. Senior and R. H. Jones (Pacific Northwest National Laboratory) 57

A hierarchical model was developed to describe the effective transverse thermal conductivity,  $K_{eff}$ , of a 2D-SiC/SiC composite made from stacked and infiltrated woven fabric layers in terms of constituent properties and microstructural and architectural variables. The model includes the expected effects of fiber-matrix interfacial conductance as well as the effects of high fiber packing fractions within individual tows and the non-uniform nature of 2D-fabric layers that include a significant amount of interlayer porosity. Model predictions were obtained for two versions of DuPont 2D-Hi Nicalon™/PyC/ICVI-SiC composite, one with a “thin” (0.110  $\mu\text{m}$ ) and the other with a “thick” (1.040  $\mu\text{m}$ ) PyC fiber coating. The model predicts that the matrix porosity content and porosity shape factor have a major influence on  $K_{eff}(T)$  for such a composite.

**2.5 EFFECT OF FIBER PROPERTIES ON NEUTRON IRRADIATED SiC/SiC COMPOSITES** - T. Hinoki, A. Kohyama and Y. Katoh (Kyoto University), L.L. Snead (Oak Ridge National Laboratory) 64

The use of SiC/SiC composites for nuclear application has recently been considered because of intrinsic low activation and superior high temperature mechanical properties of SiC. The property of SiC fiber is a key issue in order to improve mechanical properties of SiC/SiC composites following irradiation. SiC/SiC composites reinforced with unidirectional fibers were fabricated by chemical vapor infiltration method. Low oxygen and highly crystalline fibers or just low oxygen fibers were used in the composites. The specimens were irradiated at Japan Material Testing Reactor and High Flux Isotope Reactor. The effects of neutron irradiation on mechanical properties were examined by three points flexural test. Microstructure and fracture behavior were observed by scanning electron microscopy before and after neutron irradiation. The SiC/SiC composites reinforced with a low oxygen content, near-stoichiometric atomic composition, and highly crystalline SiC fibers showed the excellent stability to neutron irradiation. The mechanical property of the composites did not degrade, even after neutron irradiation up to 10 dpa, while the other materials reinforced with non-highly crystalline SiC fibers degraded significantly.

**2.6 THE EFFECT OF HIGH DOSE/HIGH TEMPERATURE IRRADIATION ON HIGH PURITY FIBERS AND THEIR SILICON CARBIDE COMPOSITES** - T. Hinoki and L. L. Snead (Oak Ridge National Laboratory), Y. Katoh, T. Nozawa and A Kohyama (Kyoto University), A. Hasegawa (Tohoku University) 74

Silicon carbide composites were fabricated by chemical vapor infiltration method with high purity fiber, Hi-Nicalon Type-S and Tyranno SA and non-high purity fiber Hi-Nicalon. SiC/SiC composites, bare fibers and CVD SiC were irradiated at 7.7 dpa and 800 °C or 6.0 dpa and 300 °C. The density of fiber and CVD SiC was measured by gradient column technique. Mechanical properties of the composites were evaluated by four-point flexural tests. Fracture surfaces were observed by SEM. Tyranno SA fiber and CVD SiC showed similar swelling behavior following irradiation at 7.7 dpa and 800 °C. Mechanical properties of Hi-

Nicalon Type-S samples and Tyranno SA samples were stable even following neutron irradiation at 7.7 dpa and 800 °C. Fracture surfaces of these samples following irradiation were similar to those of unirradiated samples with relatively short fiber pull-out.

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Russian ferritic/martensitic steels EP-450 and EP-823 were irradiated to 20-60 dpa in the BN-350 fast reactor in the form of pressurized creep tubes and small rings used for mechanical property tests. Data derived from these steels serves to enhance our understanding of the general behavior of this class of steels. It appears that these steels exhibit behavior that is very consistent with that of Western steels. Swelling is relatively low at high neutron exposure and confined to temperatures <420°C, but may be camouflaged somewhat by precipitation-related densification. The irradiation creep studies confirm that the creep compliance of F/M steels is about one-half that of austenitic steels, and that the loss of strength at test temperatures above 500°C is a problem generic to all F/M steels. This conclusion is supported by post-irradiation measurement of short-term mechanical properties. At temperatures below 500°C both steels retain their high strength ( $\sigma_{0.2}$ =550-600 MPa), but at higher test temperatures a sharp decrease of strength properties occurs. However, the irradiated steels still retain high post-irradiation ductility at test temperatures in the range of 20-700°C.

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An attempt is being made to alter the starting microstructure of CuCrZr to produce a microstructure that is not as sensitive to overageing under irradiation as the prime aged condition. Different overageing conditions (600, 700 and 800°C for 4 hours) have been examined in an earlier report, and in this report shorter overageing times at 600°C have been examined. Overageing times of 1 and 2 hours resulted in a microstructure that is similar in overall precipitate size and density to that of the oxide dispersion in GlidCop Al25. These samples will be included in future irradiation experiments to examine the effects of irradiation on the mechanical and physical properties.

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	Studies on the temperature dependence of the thermal conductivity of isotopically enriched (0.1% $^{13}\text{C}$ ) diamond [1-5] have determined the form for the intrinsic scattering phonon relaxation times in diamond. In addition, low temperature thermal conductivity measurements and infrared spectra of lightly neutron-irradiated type IIa natural diamond [6] have determined the size and concentration of extended regions of disordered carbon responsible for phonon scattering. These results have been used to model the thermal conductivity changes expected in neutron irradiated diamond at higher temperatures, from 100 to 1000K. It was found that upon irradiation to a fluence of $4.5 \times 10^{22}$ neutrons $\text{m}^{-2}$ the thermal conductivity of natural diamond went from 2200 to 370 W/mK at 300K while the thermal conductivity of diamond with a 0.1% concentration of $^{13}\text{C}$ went from 3000 to 370 W/mK at 300K.	
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We propose a comprehensive mechanism for the formation and growth of <100> interstitial loops in  $\alpha$ -Fe. This mechanism, which involves the formation of <100> junctions in the direct reaction between mobile <111> loops, reconciles long-standing experimental observations of these defects in irradiated ferritic materials with recent atomistic simulations of collision cascades and defect cluster properties in Fe, in which highly-mobile <111> clusters are seen to be the dominant feature. The <100> junctions, although metastable, grow into visible <100> loops as a consequence of the high kinetic barrier associated with rotation into <111> configurations and a very low mobility. Finally, the atomic character of <100> and  $1/2$  <111> loops is investigated with molecular dynamics simulations and the atomic configurations are used to calculate the defect image contrast through direct simulation of TEM images. The simulated images are subsequently compared with actual TEM micrographs of irradiated ferritic materials. Excellent agreement between the experiments and the simulations is found, allowing for a direct identification of the nature and structure of interstitial loops. Hence, this work provides one of the necessary links to unify simulation with experiments in  $\alpha$ -Fe and ferritic alloys subject to high-energy particle irradiation.

- 9.2 NEUTRON-INDUCED SWELLING AND EMBRITTEMENT OF PURE IRON AND PURE NICKEL IRRADIATED IN THE BN-350 AND BOR-60 FAST REACTORS** - N. I. Budylnkin, E. G. Mironova and V. M. Chernov (Bochvar Institute of Nonorganic Chemistry, Moscow, Russia), V. A. Krasnoselov (Research Institute of Atomic Reactors, Dimitrovgrad, Russia), S. I. Porollo (Institute of Physics and Power Engineering, Obninsk, Russia), and F. A. Garner (Pacific Northwest National Laboratory) 153

Pure iron and nickel were irradiated to very high exposures in two fast reactors, BOR-60 and BN-350. It appears that both nickel and iron exhibit a transient-dominated swelling behavior in the range of 2 to  $15 \times 10^{-7}$  dpa/sec, with the shortest transient at  $\sim 500^\circ\text{C}$  in nickel, but at  $< 350^\circ\text{C}$  for iron. It also appears that the duration of the transient regime may be dependent on the dpa rate. When the two metals are irradiated at  $345\text{-}355^\circ\text{C}$ , it is possible to obtain essentially the same swelling level, but the evolution of mechanical properties is quite different. The differences reflect the fact that iron is subject to a low-temperature embrittlement arising from a shift in ductile-brittle transition temperature, while nickel is not. Nickel, however, exhibits high temperature embrittlement, thought to arise from the collection of helium gas at the grain boundaries. Iron generates much less helium during equivalent irradiation.

- 9.3 THE EFFECTS OF ONE-DIMENSIONAL MIGRATION OF SELF-INTERSTITIAL CLUSTERS ON THE FORMATION OF VOID LATTICES** – H. L. Heinisch (Pacific Northwest National Laboratory) and B. N. Singh (Risø National Laboratory, Denmark) 161

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Operational difficulties were experienced with some instrumented materials irradiation experiments performed in HFIR during the two-year period preceding the outage for the beryllium changeout and reactor upgrades. This paper provides a detailed description of the operational problems, the investigations into the cause, and the proposed changes to prevent recurrence of the problems.