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1. VANADIUM ALLOYS

1.1 A SEARCH FOR CHANNEL DEFORMATION IN IRRADIATED VANADIUM TENSILE SPECIMENS - D. S. Gelles, M. B. Toloczko and R. J. Kurtz (Pacific Northwest National Laboratory **1**

A miniature tensile specimen of V-4Cr-4Ti which had be irradiated in the 17J test at 425°C to 3.7 dpa was mechanically polished, deformed to 3.9% strain at room temperature, and examined by scanning and transmission electron microscopy in order to look for evidence of channel deformation. It was found that uniform deformation can occur without channel deformation, but evidence for channeling was found with channels appearing most prominently after the onset of necking. The channeling occurs on wavy planes with large variations in localized deformation from channel to channel.

2. CERAMIC COMPOSITE MATERIALS

2.1 THE ELECTRICAL CONDUCTIVITY OF SiC$_f$/SiC WITH CORRECTIONS FOR CONTACT RESISTANCE—G. E. Youngblood and E. Thomsen (Pacific Northwest National Laboratory) and R. J. Shinavski (Hyper-Therm HTC, Inc.) **6**

Electrical conductivity (EC) data for several forms of two-dimensional, silicon carbide composite made with a chemical vapor infiltration matrix (2D-SiC/SiC) were acquired from RT to 800°C and analyzed. When using a 2-probe method, measured transverse EC-values require a fairly substantial correction due to contact resistance at the sample-electrode interface. Previously reported transverse EC-values were o too low because this correction was not considered. In this report, new transverse EC-values are reported for several forms of 2D-SiC/SiC. Many of these new values exceed the limit of 20 S/m desired for the flow channel insert application, especially at higher temperatures. The analysis showed that the EC for 2D-SiC/SiC strongly depends on the net thickness of the pyrocarbon fiber coatings, the interconnectivity and alignment of the carbon fiber network, and the thickness and, depending upon temperature, the type of composite seal coat. The in-plane EC-values also depend upon the carbon network interconnectivity, and typically greatly exceed transverse values by factors of roughly x15 (high temperatures) up to x100 (high temperatures).

2.2 MECHANICAL PROPERTIES OF ADVANCED SIC FIBER COMPOSITES IRRADIATED AT VERY HIGH TEMPERATURES – Y. Katoh, K. Ozawa, L.L. Snead (Oak Ridge National Laboratory), T. Hinoki (Kyoto University), Y.B. Choi (Hiroshima University), A. Hasegawa (Tohoku University) **17**

Six different composite materials with various near-stoichiometric silicon carbide (SiC) fiber reinforcements and pyrolytic carbon or SiC/pyrolytic carbon multilayer interphases were neutron-irradiated to ~6x10^{25} n/m2 (E > 0.1 MeV) at nominal temperatures of 800ºC and 1300ºC, and tested for tensile properties at room temperature. Only insignificant or very minor modifications to the tensile strength were admitted in all materials. However, two-parameter Weibull statistical analysis on relatively large specimen populations revealed minor but significant strength degradation for some composites. 50 – 150 nm appeared to be within the optimum PyC interphase thickness range for both fiber types in terms of tensile properties. The misfit stresses present in the unirradiated samples were significantly reduced after irradiation. The change in misfit stress was attributed to the irradiation-induced modification of coefficient of thermal expansion. True matrix cracking stress estimated from the proportional limit stress and misfit stress did not appear to degrade by neutron irradiation.

2.3 FLEXURAL TESTS OF 3C-SIC AFTER HIGH TEMPERATURE NEUTRON IRRADIATION - 26
S. Kondo, Y. Katoh, and L.L. Snead (Oak Ridge National Laboratory)

The flexure strength of miniature disk specimens were evaluated for both the unirradiated and irradiated CVD SiC by equibiaxial flexural tests developed in this work. The results for the unirradiated specimens indicated no stress magnification at the loading point, which is often concerned in biaxial tests for the disk specimens. The irradiation strength was retained after the irradiation at 1100 and 1300˚C, ~20% of reduction was observed for the samples irradiated at 1500˚C. It is clearly seen that the smooth cleavage of large grains were frequently observed in the sample irradiated at 1500˚C comparing to specimens irradiated at lower temperatures. A substantially lower population of finer defect clusters such as loops, vacancy, and vacancy clusters may be attributed to the reduction of the strength at the higher irradiation temperatures.

2.4 EFFECT OF NEUTRON IRRADIATION ON FRACTURE RESISTANCE OF ADVANCED SIC/SIC COMPOSITES—K. Ozawa, Y. Katoh, and L.L. Snead (Oak Ridge National Laboratory), and T. Nozawa (Japan Atomic Energy Agency) **34**

In order to identify the neutron irradiation effects on fracture resistance of advanced SiC/SiC composites, unloading-reloading single edge notched bend tests were conducted and an analytical model based on non-linear fracture mechanics was applied. As a result of the analysis, energy release rate contributed by macro-crack initiation of 3.1 kJ/m^2 for both unirradiated and irradiated advanced SiC/SiC composites (Hi-Nicalon Type-S (0°/90° plain woven) / multilayer / chemically vapor infiltration) is estimated. This result indicates no significant degradation in fracture resistance after neutron irradiation to 5.9×10^{25} n/m^2 at 800°C.

2.5 LOW ACTIVATION JOINING OF SiC/SiC COMPOSITES FOR FUSION APPLICATIONS - 42
C. H. Henager, Jr., D. J. Edwards, and A. L. Schemer-Kohrn (Pacific Northwest National Laboratory)

The use of SiC composites in fusion environments may require joining of plates using reactive joining or brazing. One promising reactive joining method is the use of solid-state displacement reactions between Si and TiC to produce Ti3SiC2 + SiC. We continue to explore the processing envelope for this type of joint for the Titan collaboration to produce the best possible joints to undergo irradiation studies in HFIR. Joining pressure appears to require almost 30 MPa at 1673K in order to produce strong and dense joints. Recently pressures of 40 MPa and temperatures of 1698K have been used to produce excellent joints with apparent optimal density and microstructure. This increased temperature and pressure appears to provide for better bonding between the SiC and joining compound due to interfacial SiC formation and growth during the joint processing. Electron backscatter diffraction was used to show that local epitaxy exists between the SiC and interfacial SiC grown in the joint region.

2.6 STABILITY OF 3-D CARBON FIBER COMPOSITE TO HIGH NEUTRON FLUENCE - **47**
L. L. Snead, Y. Katoh, and K. Ozawa. (Oak Ridge National Laboratory)

The dimensional stability, elastic modulus, and flexural strength of a high quality, three-dimensional balanced weave carbon fiber composite has been evaluated over a range of neutron fluence to ~32 dpa at ~ 800°C. Results indicate that while the composite exhibits continuous strengthening over this dose range, this occurs with measurable loss of mass, increased volume, and for the highest dose studied, a large reduction in elastic modulus. While the balanced weave composite was orthogonally isotropic, a significant anisotropic dimensional change occurred under irradiation. Dimensional change was dominated by fiber dimensional change and the overall shrinkage or swelling in a direction was determined by the extent to which intrinsic the fiber shrinkage was capable of restraining swelling of matrix and fiber bundles.

3.0 FERRITIC/MARTENSITIC STEELS

3.1 On the Fracture Toughness of Irradiated F82H: Effects of Loss of Constraint and Strain Hardening Capacity – T. Yamamoto, G. R.Odette (University of California Santa Barbara) and M. A. Sokolov (Oak Ridge National Laboratory). **52**

Constraint loss adjustments, based on finite element (FE) stress analysis and local critical stress, σ*, critical stressed volume, V*, cleavage criteria were applied to fracture toughness data from small pre-cracked bend bars of the IEA heat of F82H irradiated to 6.6 dpa at 300 ºC in the High Flux Isotope Reactor. The 100 MPa√m master curve method reference temperature shifts were evaluated based on both the measured toughness (ΔTmo) and after size-adjusting the toughness to small scale yielding conditions at reference specimen size (ΔTo) with values of ≈148 and 205ºC, respectively. The prediction ΔTo= CoΔ<σfl >, where Co≈ 0.68 and Δ<σfl > is the change in the average flow stress over 0-10% strain is in excellent agreement with ΔTo. The FE analyses also demonstrate an upper-bound KJc that can be measured with these small bend bars for irradiated alloys that suffer severe loss of strain hardening.

3.2 He Transport and Fate of Tempered Martensitic Steels: Summary of Recent TEM Observations – D. J. Edwards, R. J. Kurtz (Pacific Northwest National Laboratory, Richland, WA 99336, USA), G. R. Odette and T. Yamamoto (University of California, Santa Barbara, CA 93106-5070, USA) **59**

As an extension of prior work [1-3], we summarize recent observations made on He-implanted tempered martensitic steels (TMS) irradiated in the HFIR. This work is being conducted jointly between researchers at PNNL and UCSB to investigate the effects of high He levels on the microstructural evolution of irradiated TMS alloys of interest to the fusion community. He implantation occurs using a novel implantation technique whereby He is introduced via transmutation of Ni in a few micron thick Ni-Al coating applied to the surface of 3-mm TEM discs. The Ni-Al coated TEM discs of various TMS alloys were irradiated in capsules in HFIR at temperatures of 300, 400 and 500°C, and the total He levels were controlled by altering the thickness of the Ni-Al coating from 1 to 4 μm. The thickness of the coating yielded He/dpa ratios of 5 to 40. Samples were prepared from the irradiated TEM discs by a cross-sectional thinning technique, which allowed the microstructural evolution to be investigated over a range of implantation depths. The irradiation at 500°C produced relatively large, faceted cavities and smaller He bubbles that were often aligned in stringers thought to be related to the existence of pre-existing dislocations. A finer distribution of cavities were present on precipitate interfaces and grain boundaries. Cold work prior to irradiation leads to a comparable density of cavities, but at a reduced size. A lower He/dpa ratio led to a more random distribution and lower density of cavities in the matrix, but had a less noticeable effect on the bubbles/voids on the lathe and precipitate boundaries. Irradiation at 400°C produced fewer cavities and a smaller size distribution than observed at 500°C, and the distribution was random, that is, no stringers were observed. In the TEM discs irradiated at 300°C no cavities could be observed that could be distinguished from artifacts from foil oxidation and remaining sub-surface ion damage from the ion-polishing. Dislocation structures were complex and varied greatly as a function of irradiation dose and temperature, and require a more thorough characterization as the next phase of work.

3.3 **Characterization of Y Distributions and Nano-feature Formation In As Atomized, Milled, Milled and Annealed and Milled and Consolidated 14YWT Powders -** **69**
N. J. Cunningham, Y. Wu, G. R. Odette and T. Yamamoto (University of California Santa Barbara)

Reported here is work supported by the DOE Office of Nuclear Energy that is complementary to our OFES work on nano-structured ferritic alloys (NFA) for fusion applications. Three variants of Fe14Cr3W0.4Ti0.2Y rapidly solidified powders were gas atomized by Crucible Research (CR) in Ar, Ar/O and He atmospheres. This work is part of an collaborative effort between CR, LANL, ORNL, UCSB, UC Berkeley and South Dakota School of Mines to develop larger heats of NFA using best practice processing routes. One objective was to determine if rapid solidification could introduce a uniform distribution of Y prior to mechanically alloying (MA), which is the standard method for mixing Y in NFA powders. The Y distribution in the powders was characterized by TEM, EPMA, APT and SANS in as atomized, ball milled and milled and annealed conditions. It was found that pre-alloying Y during gas atomization does not produce a uniform Y distribution. However, sufficient ball milling improves the homogeneity of the Y distribution. A bimodal grain size distribution was observed in the milled and annealed powders for all gas atomization atmospheres, and Y-Ti-O precipitates are present in both the large and small grains in sizes and quantities comparable to other NFA.

3.4 **Helium Transport, Fate and Management in Nanostructured Ferritic Alloys: In Situ Helium Implanter Studies –** G. R. Odette, P. Miao, T. Yamamoto (University of California Santa Barbara, Santa Barbara), D. J. Edwards, R. J. Kurtz (Pacific Northwest National Laboratory) and H. Tanigawa (Japan Atomic Energy Agency) **74**

High helium levels produced in fusion neutron spectra may lead to severe increases in the brittle fast fracture temperature, enhanced void swelling and degradation of creep rupture properties at lower, intermediate and higher irradiation temperatures, respectively. Thus it is important to develop structural alloys with stable microstructures that can manage helium based on understanding of its transport, fate and consequences. We report on the initial results of a study of helium f in a nanostructured ferritic alloy (NFA), MA957, that is dispersion strengthened by an ultra-high density of nm-scale Y-Ti-O nanofeatures (NF). An in situ helium implanter technique uniformly deposited ≈ 380 appm helium to ≈ 6 μm in MA957 irradiated in the High Flux Isotope Reactor (HFIR) to ≈ 9 dpa at 500°C. Through focus transmission electron microscopy (TEM) showed that helium is in extremely fine bubbles that often appear to coincide with bright field features taken as NF.

4. **COPPER ALLOYS**

No contributions this period

5. **REFRACTORY ALLOYS**

No contributions this period

5. **AUSTENITIC STAINLESS STEELS**

No contributions this period

7. **FUNCTIONAL MATERIALS – INCLUDING MHD INSULATORS, COATINGS, INSULATING CERAMICS, AND OPTICAL MATERIALS**

7.1 **COMPATIBILITY OF MATERIALS EXPOSED TO ISOTHERMAL Pb-Li** – B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory, USA) **82**

The specimens from a recent set of capsule experiments are being characterized. The experiments examined the effect of Fe and Ni impurities on the 700°C dissolution of type 316 stainless steel (316SS) and Fe-9Cr-2W (T92) in Pb-Li. The addition of 1000 ppma Fe or Ni did not appear to significantly change the amount of dissolution or the surface morphology. Also, a series of capsule exposures at 600°C examined possible dissimilar material reactions between Fe and SiC. Based on surface analysis of the capsules, no Fe or Si was transferred. Future characterization will focus on C transfer. A new series of experiments planned for 500-700°C will clarify any dissimilar material interaction in this system.

8.0 **BREEDING MATERIALS**

No contributions this period

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

9.1 **MODELLING THERMODYNAMICS OF ALLOYS FOR FUSION APPLICATION -** P. Erhart, M. Serrano de Caro, B. Sadigh (Lawrence Livermore National Laboratory), S.G. S. Srivilliputhur (UNT), J. Hetherly (UNT), and A. Stukowski (Germany) **88**

The atomic scale description of bcc ferromagnetic alloys has reached the barrier of the finite temperature behavior of the magnetic excitations, with current models valid only for temperatures well below the Curie temperature (~ 1000 K). A description for higher temperatures, valid for T < 1000 K, has made significant progress in the last year, with development of both tools and algorithms.

- The composition dependent embedded atom model for ternary alloys (CD-EAM) is in Lammps, a freely distributed code for molecular dynamics. Also implemented is a ‘variance constrained’ Metropolis Monte Carlo, MMC, algorithm in Lammps, for use with ternary alloys.
- A ‘one site’ modified version of the CD-EAM significantly improves the performance of a MMC algorithm without affecting the accuracy of the interatomic potential.
- A ternary FeCr-He potential was validated against *ab initio* data on He in FeCr.
- Simulation of He bubbles in FeCr alloys has started, determining the pressure versus size relation for the 1He/vacancy ratio and the mechanisms of bubble growth at varying density.

These tools are free for distribution, and some of them are already part of the Lammps official download package, some others are at the Stukowski web page, and the parallel variance constrained Metropolis Monte Carlo within Lammps will soon be released.

9.2 **A NEW Fe-He INTERATOMIC POTENTIAL BASED ON *Ab Initio* CALCULATIONS IN α-Fe** – F. Gao, H. L. Heinisch, R. J. Kurtz (Pacific Northwest National Laboratory) **97**

A new interatomic potential for the Fe-He interaction has been developed by fitting to results obtained from *ab initio* calculations. Based on electronic hybridization between Fe d-electrons and He s-electrons, an s-band model, along with a repulsive pair potential**,** has been proposed to describe the Fe-He interaction. The atomic configurations and formation energies of single He defects and small interstitial He clusters are considered in the fitting process. The binding properties and relative stabilities of the He-vacancy and interstitial He clusters are studied, and the results are compared with available *ab initio* calculations as well as with those obtained from previous Fe-He potentials. The present Fe-He potential overcomes the disadvantages that appear in previous Fe-He potentials, and it will be applied to simulate the interactions of He with Fe in various microstructural features in large-scale molecular dynamics simulations.

9.3 **ATOMISTIC STUDIES OF PROPERTIES OF HELIUM IN BCC IRON -** **104**
David Stewart, Yuri Osetskiy, and Roger Stoller (Oak Ridge National Laboratory)

Helium created by transmutations plays an important role in the response of reduced-activation ferritic/martensitic (RAFM) steels to neutron radiation damage. We have performed extensive atomistic simulations using the ORNL 3-body Fe–He interatomic potential combined with three interatomic potentials for the iron matrix. Interstitial helium is very mobile and coalesces to form interstitial clusters, with some mobility of small clusters. When an interstitial He cluster reaches sufficient size, it punches out an Fe interstitial, creating an immobile helium–vacancy cluster. If more helium atoms join it, more Fe interstitials can be created, and the He–V defect is a nascent bubble. These mechanisms are investigated in simulations that examine the nucleation of He defects. Mobile interstitial He clusters and helium bubbles 1 to 6 nm diameter are also simulated separately. Results are compared based on temperature and interatomic potentials used.

9.4 **MULTIPOLAR ELASTIC REPRESENTATION OF DEFECT FIELDS IN METALS-** D. Seif (University of California, Los Angeles) and N. M. Ghoniem (University of California, Los Angeles) **113**

In this study, a hybrid method has been developed to couple the results of molecular statics calculations with classical isotropic elasticity theory to extract a continuum representation of various defects through the use of force multipoles. These calculations have provided a means to account for the changes in defect energetics due to local strain environment effects. In uniform strain environments, the use of the elastic dipole tensor is sufficient to calculate the interaction energy. However we find the use of higher order multipole tensors necessary to represent many defects and their interactions in environments with strain gradients, such as the vicinity of grain boundaries or other extended defects. In addition, isotropic elastic Green's functions are found to provide material response very consistent with atomistic simulations when these higher order terms are implemented. We have made calculations of interactions between defects including Fe and He point defects, Fe self-interstitial clusters, He-vacancy clusters, and extended defects in α-iron.

10. **DOSIMETRY, DAMAGE PARAMETERS, AND ACTIVATION CALCULATIONS**

No contributions this period

11. **IRRADIATION FACILITIES AND TEST MATRICES**

No contributions this period

12. **HFIR Irradiation Experiments –** F. W. Wiffen, Oak Ridge National Laboratory **123**

The current status of Fusion Materials Program irradiation experiments in the HFIR reactor is tabulated.