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1. FERRITIC/MARTENSITIC STEELS

See also items 6.1, 6.2, and 6.3.

1.1 TEM OBSERVATION OF DUAL ION BEAM IRRADIATED F82H MOD.3 AND MA957 – **1**
T. Yamamoto, Y. Wu, G. R. Odette (University of California Santa Barbara), K. Yabuuchi, A. Kimura (Kyoto University)

Microstructures of tempered martensitic steel F82H mod.3 and NFA MA957 have been characterized after dual ion beam (Fe^{3+} and He^{+}) irradiation to a nominal condition of 10 dpa and 400 appm He at ≈ 480°C. The irradiations were performed at dual beam facility, DuET, located at Kyoto University in Japan. Helium bubbles were found at depths greater than 300 nm in F82H mod.3. The average bubble diameter was larger and the number density comparable to or lower than for those observed following in situ He implanter (ISHI) irradiations in the HFIR JP26 experiment at 500°C at similar dpa and He levels (9 dpa and 380 appm He), but at much lower dpa rates. A few larger cavities, that are likely voids, were observed following the DuET irradiation. However, a bimodal bubble and void size distribution was less apparent in the DuET case, compared to the ISHI results. No He bubbles were observed in MA957 following DuET irradiation to a condition similar to that for the JP26 ISHI experiment where bubbles were observed. Bubbles were only visible at DuET conditions of > 15 dpa and He > 550 appm, and they were less numerous than found in the ISHI irradiation at lower damage and He levels.

1.2 FURTHER ATOM PROBE TOMOGRAPHY STUDIES OF NANOSTRUCTURED FERRITIC ALLOY MA957 IN THREE CONDITIONS – **11**
Nicholas J. Cunningham, G. Robert Odette and Erich Stergar, University of California – Santa Barbara

We report on atom probe tomography (APT) studies on nano-structured ferritic alloys (NFAs) that contain an ultrahigh density of nm-scale Y-Ti-O nanofeatures (NFs). A local electrode atom probe (LEAP) was used to characterize the NFs in both as-extruded bar (US) and thick walled tube (French) heats of MA957. APT was also carried out on the US MA957 following long-term thermal aging (LTTA) at 1000ºC for 19 kh. The as-extruded US MA957 was found to contain ≈ $3.2x10^{23}$ NFs/m^3 with an average diameter of ≈ 2.4 nm. The French MA957 tubing contained an average of $6.1x10^{23}$ NFs/m3 with an average diameter of ≈ 2.8 nm. The average Y/Ti/O ratio of ≈13/47/40 was similar in both heats. The NFs coarsen under 1000°C LTTA, with average number densities and diameters decreasing to $7x10^{22}$ NFs/m^3 and increasing to ≈ 2.8 nm, respectively. The NF composition in the aged condition is relatively unchanged with only a slight increase in the Y/Ti ratio.

2. CERAMIC COMPOSITE STRUCTURAL MATERIALS

2.1 TITAN TASK 2-3 SILICON CARBIDE BEND STRESS RELAXATION CREEP STUDY: PHASE-II EXPERIMENT – **17**
Y. Katoh, K. Ozawa (Oak Ridge National Laboratory), and T. Hinoki (Kyoto University)

A study of irradiation creep behavior of silicon carbide ceramics and composites is part of Task 2-3 of the US/Japan TITAN collaboration on fusion materials and blanket technology. In the Phase-I experiment, low fluence irradiation creep behavior of monolithic silicon carbide ceramics was

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studied using the bend stress relaxation (BSR) technique. The objective of the Phase-II experiment is to gain understanding of the stress relaxation and creep behavior of silicon carbide ceramics, fibers, and composites under neutron irradiation to higher fluences at elevated temperatures. The neutron irradiation will be performed using the fixed rabbit facilities of the High Flux Isotope Reactor. The Phase-II program will irradiate 13 rabbit capsules; 8 for monolithic and composite samples and 5 for fiber samples. Target irradiation temperatures are 300, 500, 800, and 1200°C. The present schedule assumes the initiation of irradiation in early 2011.

2.2 LOW ACTIVATION JOINING OF SiC/SiC COMPOSITES FOR FUSION APPLICATIONS – 25

C. H. Henager, Jr., R. J. Kurtz (Pacific Northwest National Laboratory, Richland, WA 99336, USA), and M. Ferraris, (Politecnico di Torino, Torino, Italy)

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 Ti_3SiC_2 + 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. The TITAN collaboration has designed miniature torsion joints for preparation, testing, and irradiation in HFIR. As part of that project PNNL synthesized 40 miniature torsion joints and several were tested for shear strength prior to irradiation testing in HFIR. The resulting tests indicated that joint fixture alignment problems cause joint strengths to be lower than optimal but that several joints that were well aligned had high shear strengths and promising mechanical properties. High joint strengths cause non-planar shear fracture and complicate strength analysis for these miniature torsion joints.

2.3 CHARACTERIZATION BY SEM OF THE PYROCARBON FIBER COATING IN 2D-SiC/CVI-SiC – 31

G. E. Youngblood (Pacific Northwest National Laboratory)

The previous report examined electrical conductivity (EC) data from RT to 800°C for several forms of two-dimensional silicon carbide composite made with a chemical vapor infiltration (CVI) matrix (2D-SiC/CVISiC), an important quantity needed for the design of an FCI. We found that both in-plane and transverse EC-values for 2D-SiC/CVI-SiC strongly depended on the total thickness of the highly conductive pyrocarbon (PyC) fiber coating and the alignment of the carbon coating network. Furthermore, the transverse EC depended on the degree of interconnectivity of this network. For our EC-modeling efforts we used either "nominal" coating thickness values provided by the composite fabricator or we made thickness estimates based on a limited number of fiber cross-section examinations using SEM. Because of the importance of using a truly representative coating thickness value in our analysis, we examined numerous new SEM cross-sectional views to reassess the reliability of our limited number of original coating thickness measurements as well as to obtain an estimate of the variation in thickness values for different composite configurations.

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2.4 EVALUATION OF DAMAGE TOLERANCE OF ADVANCED SiC/SiC COMPOSITES AFTER NEUTRON IRRADIATION – 38
K. Ozawa, Y. Katoh, L.L. Snead (Oak Ridge National Laboratory), T. Nozawa (Japan Atomic Energy Agency), T. Hinoki (Kyoto University)

The effect of neutron irradiation on damage tolerance of two nuclear grade SiC/SiC composites (plain-woven Hi-Nicalon™ Type-S fiber-reinforced, CVI SiC matrix composites with a multilayer interphase and unidirectional Tyranno™-SA3 fiber-reinforced, NITE matrix with a carbon mono-layer interphase) was evaluated by means of miniaturized single-edged notched-beam test. No significant changes in crack extension behavior and in the load-loadpoint displacement characteristics such as the peak load and hysteresis loop width were observed after irradiation to 5.9×10^{25} n/m^2 (E > 0.1 MeV) at 800°C and to 5.8×10^{25} n/m^2 at 1300°C. The global energy balance analysis based on non-linear fracture mechanics estimated the energy release rate contributed by macro-crack initiation to be 3±2 kJ/m2 for both the unirradiated and irradiated composites. The effects of neutron irradiation on fracture resistance of these composites appeared insignificant for the conditions examined.

3.0 REFRACTORY METALS AND ALLOYS

See also item 6.4.

3.1 THERMO-MECHANICAL DAMAGE OF TUNGSTEN SURFACES EXPOSED TO RAPID TRANSIENT PLASMA HEAT LOADS – 44
T. Crosby and N.M. Ghoniem (University of California, Los Angeles)

Tungsten has one of the highest melting points of any metallic material, and for this reason, it is used in applications where extreme heat fluxes and thermo-mechanical conditions are expected. Recently, international efforts have focused on the development of tungsten surfaces that can intercept energetic ionized and neutral atom and heat fluxes in the divertor region of magnetic fusion confinement devices, and as armor in chamber wall applications in inertial confinement fusion energy systems. The combination of transient heating and local swelling due to implanted helium and hydrogen atoms has been experimentally shown to lead to severe surface and sub-surface damage. The thermo-mechanical model is based on elasticity, coupled with a reaction-diffusion model of material swelling and grain boundary degradation due to helium and deuterium bubbles resulting from the plasma flux. This material state is also coupled with a transient heat conduction model for temperature distributions following rapid thermal pulses. The multi-physics model includes contact cohesive elements for grain boundary sliding and fracture. Results of the computational model are compared to experiments on tungsten bombarded with energetic helium and deuterium particle fluxes.

3.2 MECHANICAL PROPERTIES OF W-1.1%TiC ALLOY – 52
M. A. Sokolov (Oak Ridge National Laboratory)

A small disk of W-1.1%TiC alloy produced by Japanese researchers led by Hiroaki Kurishita was sent to ORNL for mechanical testing and characterization. The purpose of the study is to evaluate the effect of small additions of TiC in improving the performance of W-based materials designed for operation in the divertor environment. This is part of on-going efforts of the international fusion community to understand and improve the ductility and toughness of W-based materials using nano-scale microstructural modification. A test plan was developed to perform limited

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fracture toughness and tensile evaluation of this alloy. The results of this evaluation will be presented in the next semi-annual report.

4.0 OTHER STRUCTURAL AND SPECIAL PURPOSE MATERIALS

No contributions this period.

5. CORROSION AND COMPATIBILITY

5.1 COMPATIBILITY OF MATERIALS EXPOSED TO ISOTHERMAL Pb-Li – **54**
B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory, USA)

Initial isothermal capsule experiments were conducted to compare the behavior of oxide dispersion strengthened (ODS) Fe-Cr alloys to prior results on wrought ferritic-martensitic (FM) alloys. Also, the performance of corrosion resistant, Al-rich diffusion coatings on these alloys was investigated. To further understand the performance of these coatings in Pb-Li, several experiments are in progress including a time series of experiments and a more detailed study of the unexpectedly high Al loss observed in prior experiments. New Pb-Li was cast to eliminate the prior issue of Li composition variability. Finally, to investigate any potential dissimilar material interaction between Fe and SiC, a set of capsules with SiC inner capsules is being assembled for exposures at 500, 600 and 700°C.

6.0 THEORY AND MODELING

6.1 DIFFUSION OF He INTERSTITIALS AND He CLUSTERS IN α-Fe – **58**
H. Deng (Hunan University), F. Gao, H. L. Heinisch and R. J. Kurtz (Pacific Northwest National Laboratory)

The accumulation of He atoms in materials will significantly degrade the mechanical properties of materials; therefore, understanding the properties of He interstitials and their clusters in materials is of fundamental importance within a fusion reactor environment. The diffusion properties of single He interstitials and He clusters in the bulk and grain boundaries of α-Fe are being studied using molecular dynamics with a new Fe−He potential. It is found that the migration barrier for a single He interstitial in the bulk is very low, which is consistent with the result obtained using *ab initio* methods. Large He clusters can cause Fe self-interstitial atoms (SIA) to be formed, which can be trapped by the resulting vacancy, forming a He-vacancy complex. It is found that for He interstitials in grain boundaries (GBs), the He migration is one-dimensional in a Σ11 GB, while it is two-dimensional in a Σ3 GB at 600 K and three-dimensional at higher temperatures.

6.2 ATOMISTIC STUDIES OF PROPERTIES OF HELIUM IN BCC IRON USING THE NEW He–Fe POTENTIAL – **66**
David M. Stewart, Stanislav Golubov (Oak Ridge National Laboratory and the University of Tennessee), Yuri Ostesky, Roger E. Stoller, Tatiana Seletskaia, and Paul Kamenski (Oak Ridge National Laboratory)

We have performed atomistic simulations of helium bubble nucleation and behavior in iron using a new 3-body Fe–He inter-atomic potential combined with the Ackland iron potential. Updated results from ongoing large simulations examining the nucleation of helium defects are presented. MS simulations of the change in pressure when a void is added to a perfect crystal are used to

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estimate the pressure of an equilibrium helium bubble of the same size. This method is independent of the choice of helium potential. When an Fe interstitial encounters a helium bubble, it can recombine with one of the vacancies in the bubble, leading to a bubble with a higher He/V ratio and hence pressure. We investigate how far this process can go before the bubble will not accept any more SIAs.

6.3 A MULTI-SCALE MODEL OF HELIUM TRANSPORT AND FATE IN IRRADIATED TEMPERED MARTENSITIC STEELS AND NANOSTRUCTURED FERRITIC ALLOYS – 73
T. Yamamoto, G.R. Odette (Department of Mechanical Engineering, University of California Santa Barbara), R.J. Kurtz (Materials Science Division, Pacific Northwest National Laboratory) and B.D. Wirth (University of Tennessee, Knoxville)

Development and application of a multiscale model of the transport and fate of He in irradiated nanostructured ferritic alloys and tempered martensitic steels are described. Model predictions for He bubble average size, size distribution and number density are in reasonably good agreement with recent observations in in situ helium implanter experiments on F82H mod.3, 12YWT and MA957.

6.4 FIRST-PRINCIPLES INVESTIGATION OF THE INFLUNECE OF ALLOYING ELEMENTS ON THE ELASTIC AND MECHANICAL PROPERTIES OF TUNGSTEN – 90
G. D. Samolyuk, Y. N. Osetskiy, and R. E. Stoller (Oak Ridge National Laboratory)

The equilibrium lattice parameter, elastic constants and phonon dispersions were calculated for a set of binary $W_{1-x}Tm_x$ alloys with different transition metal, Tm, concentrations within the local density approximation of density functional theory. Reasonable agreement between results obtained using conventional super-cell and virtual crystal approximation approaches has been demonstrated. Alloying W with transition metals with larger number of d-electrons changes the symmetry of the core of a dislocation from symmetric to asymmetric and reduces the value of the Peierls barrier.

7. IRRADIATION AND EXPERIMENTAL METHODS AND ANALYSIS

7.1 IRRADIATION TEMPERATURE DETERMINATION OF HFIR TARGET CAPSULES USING DILATOMETRIC ANALYSIS OF SILICON CARBIDE MONITORS – 94
T. Hirose, N. Okubo, H. Tanigawa (Japan Atomic Energy Agency), Y. Katoh, A.M. Clark, J.L. McDuffee, D.W. Heatherly, R.E. Stoller (Oak Ridge National Laboratory)

The irradiation temperatures of the HFIR target capsules JP-26 and JP-27 were determined by dilatometric analysis of silicon carbide passive temperature monitors. The monitors from holders for SSJ3 tensile specimens demonstrated good agreement with the design temperatures derived from finite element model (FEM) analysis and were consistent with post-irradiation hardness of F82H. Although the irradiation temperatures for some bend-bar (PCCVN and DFMB) holders were higher than FEM analysis, hardness tests on irradiated F82H implied that actual irradiation temperatures were close to the design temperatures.

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7.2 ESTIMATION OF HELIUM PRODUCTION BY THE NICKEL FOIL IMPLANTER TECHNIQUE FOR BEND STRESS RELAXATION TESTS IN THE TITAN PHASE II RABBIT IRRADIATION CAMPAIGN – 100
K. Ozawa, Y. Katoh, L.L. Snead (Oak Ridge National Laboratory), T. Yamamoto (University of California, Santa Barbara), T. Hinoki (Kyoto University), A. Hasegawa (Tohoku University)

He profiles in bend stress relaxation specimens from the thin Ni foil implanter technique were calculated for the TITAN Phase II Campaign. The calculations revealed that the distribution of the implanted transmuted helium is uniform at 2.1, 15 and 21 appm He/dpa to a depth of 11 μm for the case of a 2 μm-thick implanter foil for irradiation to 1, 10 and 20×10^{25} n/m^2 (E > 0.1 MeV), equivalent to 1, 10, and 20 dpa-SiC in the HFIR-PTP. It is noted that the He/dpa ratio is strongly fluence dependent, since natural Ni was used for the implanter foil and hence the He is produced by a two neutron capture sequence.

8.0 IRRADIATION EXPERIMENTS AND TEST MATRICES

8.1 OPERATING CONDITIONS AND IRRADIATION HISTORY FOR EXPERIMENT MFE-RB-15J – 110
J. McDuffee, D. Heatherly (Oak Ridge National Laboratory)

The MFE-RB-15J experiment was designed to irradiate steel specimens at 300 and 400°C for 10 cycles in the RB* irradiation facility in HFIR. The irradiation vessel was divided into three subcapsules. The specimen regions of the upper and lower subcapsules were about 7.7 cm long, located ±14 cm from the reactor midplane, and designed to operate at 300°C. The specimen region of the middle subcapsule was about 11.5 cm long, centered at the reactor midplane, and designed to operate at 400°C.

Each subcapsule was filled with lithium, which became molten during operation and solidified during reactor outages. Thermocouples were located at the centerline of each subcapsule and extended upward through part of the axial length of the subcapsule.

Because of concerns over the potential for a volatile reaction between the lithium and water in the event of a containment failure, the specimen-containing subcapsules were housed inside two outer containments. There were small gas gaps between the primary and secondary containments and between the secondary containment and the subcapsules. The outer gas gap was filled with helium. The inner gas gap was filled with a mixture of helium and neon, and the relative concentrations of the two were controlled to provide the gas conductivity necessary to achieve the desired temperatures. The gas compositions for each of the three subcapsules were controlled separately, although not completely independently.

8.2 DESIGN OF THE JP30 AND JP31 EXPERIMENTS – 115
J. McDuffee, D. Heatherly, N. Cetiner (Oak Ridge National Laboratory)

Two experiments, JP30 and JP31, have been designed to place various stainless steel specimens in the flux trap of the High Flux Isotope Reactor (HFIR). These designs are very similar to other experiments irradiated previously in HFIR (e.g., JP26, JP27, JP28, JP29).

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The JP30 and JP31 experiments are designed to irradiate F82H specimens of various sizes and types in the flux trap of HFIR at temperatures in the range of 300 to 650°C. The specimens are typically contained within holders of either DISPAL (dispersion-strengthened aluminum) or a vanadium alloy (V-4Cr4Ti). The primary outer containment is an Al-6061 tube with an outer diameter of 1.27 cm. Helium is used as the fill gas inside the experiment. The specimen temperature is controlled by the size of the gap between the holder and housing. This report summarizes the work described in the design and analysis calculation for this project.

8.3 HFIR IRRADIATION EXPERIMENTS – December 31, 2010 – 134
F. W. Wiffen (ORNL)

Summary of recent, current, and planned Fusion Materials Program Experiments in the High Flux Isotope Reactor (HFIR).