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1. FERRITIC/MARTENSITIC STEELS

See also items 4.1, 5.1, and 8.1.

1.1 Selected Reduced Activation Ferritic-Martensitic (RAFM) Steels For HFIR Experiments JP-30/31 — 1
L. Tan (Oak Ridge National Laboratory)

Three heats of RAFM steels were selected for HFIR experiments. A modified-NF616 was also selected for comparison. Tensile tests of the heats were performed at temperatures of 23°C, 300°C, 400°C, and 650°C, which showed tensile properties comparable to the oxide dispersion strengthened (ODS) steel PM2000. Microstructural analysis indicates that fine lath boundaries with nano-precipitates had been developed in the heats by the specific thermomechanical treatment.

1.2 On the Structure and Chemistry of Complex Oxide Nanofeatures in Nanostructured Ferritic Alloy U14YWT — 5
D. Bhattacharyya, P. Dickerson, S. A. Maloy, A. Misra, M. A. Nastasi *(Los Alamos National Laboratory)* and G. R. Odette *(University of California Santa Barbara)*

The remarkable radiation damage resistance of nanostructured Ferritic alloys (NFAs) is attributed to the large numbers of matrix nano-features (NFs) of various types, which can enhance the recombination of displacement defects and trap transmutant helium in fine scale bubbles. Conventional and high resolution transmission electron microscopy and energy dispersive spectroscopy were used to characterize the various types of NF and larger oxide phases in a model 14Cr-3W-0.4Ti-0.25Y_2O_3 NFA (14YWT) Hot Isostatic Pressed (HIP-ed) at 1150°C. Large $CrTiO_3$ precipitates (50-300 nm) and small diffracting NFs (< 5 nm) were found in this alloy. One major new result is the observation of an additional type of nano-feature (10-50 nm), orthorhombic in structure, with a square center cross section, that constitutes a new kind of Y-Ti oxide phase with lattice parameters different from those of known Y and Ti complex oxides. The interfaces of these particles seem to be semicoherent, while manifesting a possible orientation relationship with the BCC matrix. The ratio of Y to Ti varies between < 1 and 2 for these larger NFs.

1.3 Transmission Electron Microscopy Characterization of the Structure and Composition of the Nanofeatures in Nanostructured Ferritic Alloy MA957 — 25
Y. Wu, E. Haney, N. Cunningham and G. R. Odette *(Univ. of California Santa Barbara)*

Bright field transmission electron microscopy (TEM), high resolution TEM (HRTEM), scanning TEM (STEM) and energy dispersive X-ray (EDX) spectroscopy were combined to comprehensively characterize the Ti-Y-O enriched nano-scale features (NF) in a nanostructured ferritic alloy (NFA), MA957. The characterization included the NF number densities, size distributions, volume fractions, crystal structures and compositions. In foil

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measurements were complemented by studies of NF on extraction replica foils. The conditions examined included: two heats of extruded MA957; one of these heats following long-term thermal aging at 1000°C for 19.53 kh; and two friction-stir-weld variants. The study revealed the presence of diffracting complex oxide phases that varied somewhat with the feature size. However, the smallest features, with diameters (d) < 5 nm, were found to be consistent with cubic pyrochlore $Y_2Ti_2O_7$ complex oxides with average Y/Ti ratios of ≈ 1. Long-term thermal aging coarsened the NF, while friction stir welding had relatively little effect on their number density and size distribution.

1.4 Inter-Laboratory Atom Probe Tomography Characterization Study of the Nanofeatures and the Bulk and Matrix Compositions of US MA957 — **41**

N. J. Cunningham, G. R. Odette (Univ. California Santa Barbara), E. Stergar (University of Leoben), E. A. Marquis (Oxford University), A. Etienne, C. Hatzoglou, B. Radiguet and P. Pareige (University of Rouen)

APT studies were carried out at the University of California Santa Barbara (UCSB), University of Leoben (UL) in Austria, Oxford University (OU) in England and University of Rouen (UR) in France. The tools used at UCSB, OU and LU were all Cameca (previously Imago) HR3000X-HR Local Electrode Atom Probes (LEAPs), while the fourth instrument at Rouen was a Camaca Laser Assisted Wide Angle Tomographic Atom Probe (LAWATAP). The measurements reported by the various APT groups are in remarkable agreement with one another. The bulk average compositions (at.%) for Fe, Cr, Ti, Y and O are: 83.6±0.39, 14.8±0.46, 0.771±0.059, 0.064±0.022 and 0.273±0.031, respectively. The corresponding matrix Fe, Cr, Ti, Y and O are, respectively: 84.1±0.283, 14.7±0.212, 0.636±0.021, 0.017±0.001 and 0.109±0.020. The average NF compositions, based only on their Y, Ti and O contents (at.%), are 13.9±0.92 Y, 41.6±3.34 Ti and 45.4±4.77 O, respectively. The corresponding average NF number density (N) and average diameter (<d>) are: $5.1\pm1.06\times10^{23}/m^3$ and 2.72±0.56 nm, respectively.

1.5 The Effect of Bulk Oxygen Concentration on the Micro-nanostructure and Strength of a 14Cr Nanostructured Ferritic Alloy — **47**

Y. Wu, N. J. Cunningham, A. Etienne, E. Haney, G. R. Odette (University of California Santa Barbara), E. Stergar (Leoben University), D. T. Hoelzer (Oak Ridge National Laboratory), B. D. Wirth (University of California Berkeley) and S. A. Maloy (Los Alamos National Laboratory)

The effects of bulk O variations on the characteristics of the grain structure, nanofeatures (NFs) and microhardness of nanostructured ferritic alloys (NFA) were investigated. The grain structures were characterized by optical metallography and transmission electron microscopy (TEM). The NFs were characterized by TEM, atom probe tomography (APT) and small angle neutron scattering (SANS). Based on bulk chemical analysis of the milled powders, the O content ranged from 0.065 to 0.249 (wt.%). The variations in O resulted from differences in the milling conditions. The low O powders (OW3) were milled with a procedure that minimized contamination, while the high O powders (OW1), that initially had a low O content in the as atomized condition, were contaminated with both N and O during milling. The intermediate O powders (OW4) minimized this contamination while increasing O with additions of FeO during milling. The three powder batches were all consolidated by hot

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isostatic pressing (HIPing) at 1150°C and 200 MPa. All the consolidated alloys had bimodal distributions of grain sizes, that were characterized by the two average large and small sizes ($d_{s,l}$) and the relative fraction of the small grains (f_s). The NFs were Y-Ti-O enriched phases characterized by their number density (N), size distributions and average diameter (<d>), composition (especially the Y/Ti ratio) and phase structure (not evaluated in this study). Both the large and small grain sizes decreased with increasing O contents, while the fraction of small grains increased. The size of the NFs decreased and their number density increased with increasing O. TEM energy dispersive x-ray (EDX) studies indicated the low O alloy contained fewer and coarser features compared to alloys with higher O. TEM showed that the low O features were much more highly enriched in Y compared to Ti, including a majority that contained no Ti. However, APT showed that NFs in the low O alloy with <d> ≈ 5 nm had Y/Ti ratios < 1 that is only slightly larger than found in the other cases. The Vickers microhardness (H_v) of the alloys increased significantly with increasing O, reflecting both the effects of differences in the grain size and scale dispersion strengthening features. The H_v increased systematically with the volume fraction of NFs measured by SANS.

1.6 Effects of Neutron Irradiation on Hardness Properties of Friction Stir Processed ODS Ferritic Steel — **58**

Sanghoon Noh (Kyoto University), M. A. Sokolov (Oak Ridge National Laboratory), Ryuta Kasada, Akihiko Kimura (Kyoto University) and Takuya Nagasaka (National Institute for Fusion Science)

Oxide dispersion strengthened ferritic steels (ODS-FS) have been considered as one of the most promising structural materials for advanced nuclear systems such as fusion reactors and next generation fission reactors, because of its excellent elevated temperature strength, corrosion and radiation resistance. Especially, irradiation resistance is a critical issue for the high performance of ODS-FS. In this study, effects of the irradiation on hardness properties of friction stir processed (FSP) ODS-FS were investigated. FSP technique was employed on ODS-FS. A plate specimen was cut out from the cross section and irradiated to 1.2 dpa at 573K in the High Flux Isotope Reactor (HFIR). To investigate the effect of neutron irradiation on processed area, the hardness distributions were evaluated on the cross section. Hardness of FSP ODS-FS was various with each microstructure after irradiation to 1.2 dpa at 573K. The increase of Vickers hardness was significant in the stirred zone and heat affected zone. Base material exhibited the lowest hardening about 38HV. Since nano-oxide particles in stirred zone showed identical mean diameter and number density, it is considered that hardening differences between stirred zone and base material is due to differences in initial dislocation density.

1.7 Joining of 14YWT and F82H by Friction Stir Welding — **62**

D. T. Hoelzer, M. A. Sokolov and Z. Feng, ORNL

A sample was prepared for the initial joining study using friction stir welding (FSW) that consisted of 4 specimens of the advanced oxide dispersion strengthened (ODS) 14YWT-SM6 ferritic alloy, 15 mm long, 6 mm wide and 2.86 mm thick each, that were spot welded in a corresponding slot that was machined in an F82H plate. The FSW run was successfully performed on the sample using a polycrystalline boron nitride tool (PCBN) that resulted in

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joints showing good bonding between specimens of 14YWT-SM6 and between 14YWT-SM6 and F82H. The joints were characterized by light microscopy and SEM analysis and were observed to be relatively narrow in width. However, porosity was observed in the thermomechanically affected zone (TMAZ) of 14YWT-SM6 on the advancing side of the FSW joint and at the interface between F82H and 14YWT-SM6. Further refinements in the FSW process will be required to minimize defects including porosity.

2. CERAMIC COMPOSITE STRUCTURAL MATERIALS

See also items 5.1 and 8.2.

2.1 Tensile Properties of Unidirectional CVI SIC/SIC Minicomposites with a Multilayer interphase and Near-Stoichiometric SIC Fibers — **76**
K. Ozawa, Y. Katoh, L. L. Snead (Oak Ridge National Laboratory), T. Nozawa, H. Tanigawa (Japan Atomic Energy Agency)

Tensile properties of unidirectional single tow SiC/SiC minicomposites reinforced with Hi-Nicalon™ Type-S and Tyranno™-SA3 were evaluated. The interphase structure was pyrolytic carbon (PyC) / SiC multilayer and the thickness of the innermost PyC layer was varied (~70, ~240 and ~1150 nm). Only the Hi-Nicalon Type-S minicomposites with ~240 and ~1150 nm-thick PyC interphases exhibited the pseudo-ductile behavior; others reinforced with Tyranno-SA3 and the Hi-Nicalon Type-S composite with 70 nm-thick PyC interphase underwent brittle to near-brittle fracture behavior. The brittle failure behavior for those composites was attributed primarily to the quite low fiber volume fraction (5-10%). The Hi-Nicalon Type-S minicomposites with ~240 nm-thick PyC interphase exhibited ultimate tensile strength equivalent to 79-104% of the fiber bundle strength but only 70-85% of the strength for the ~1150 nm interphase for a given gauge length. Based on the analysis of the hysteresis loops recorded during the tensile loading and unloading sequences, the sliding stress estimated for the ~1150 nm-thick interphase appeared to be about 0.7 times that of the ~240 nm-thick interphase. This shows that both composites satisfied the global load sharing condition, but the composite with the thicker interphase could not achieve its fiber bundle strength, probably due to the lower interfacial sliding stress.

2.2 Low Activation Joining of SiC/SiC Composites for Fusion Applications — **87**
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 likely requires joining of plates using reactive joining or brazing. One promising reactive joining method uses solid-state displacement reactions between Si and TiC to produce Ti_3SiC_2 + SiC. We continue to explore the processing envelope for this joint for the TITAN collaboration in order to produce optimal joints to undergo irradiation studies in HFIR. The TITAN collaboration has designed miniature torsion joints for preparation, testing, and irradiation in HFIR. PNNL synthesized 40 miniature torsion joints and several were tested for shear strength prior to irradiation testing in HFIR. The resulting tests indicated that 1) joint fixture alignment problems cause joint strengths to be lower than optimal, 2) that non-planar torsion test failures may limit th

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the effectiveness of the miniature specimen design, and 3) that several joints that were well aligned had high shear strengths and promising mechanical properties. In summary, it appears that high joint strengths cause non-planar shear fracture and complicate strength analysis for miniature torsion specimens.

3. REFRACTORY METALS AND ALLOYS

See also items 5.2 and 6.1.

3.1 Thermomechanical Damage of Tungsten Under Transient Plasma Heat Loads — **96**
T. Crosby and N. M. Ghoniem (University of California, Los Angeles)

Tungsten is a primary candidate for plasma facing components (PFCs) in fusion energy systems because of its superior thermophysical properties. International efforts are focused on the development of tungsten surfaces that can intercept ionized atoms and high heat flux in the divertor region of magnetic fusion confinement devices. Designing a robust interface between thermonuclear plasma and the heat sink material in the divertor remains a major challenge to the success of future fusion power reactors. In this report, we present a multiphysics computational model to determine the extent of thermomechanical damage in tungsten under type-I edge localized modes (ELMs). The model is formulated in a finite element framework, and is based on a multiphysics approach that combines elastoplastic mechanical analysis and thermal heat conduction.

The model is coupled with a reaction-diffusion model of material swelling and grain boundary degradation due to helium bubbles resulting from the plasma flux. Contact cohesive elements are used to model grain boundary sliding and fracture. The present results establish the connection between plastic deformation and the onset of material damage.

3.2 Development of Mo-Re Alloys — **105**
M. A. Sokolov and E. K. Ohriner (Oak Ridge 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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4. OTHER STRUCTURAL AND SPECIAL PURPOSE MATERIALS

4.1 Effects of Neutron Irradiation on Hardness of W-ODS Ferritic Steel Joints — **111**
Sanghoon Noh (Kyoto University), M. A. Sokolov (Oak Ridge National Laboratory), Ryuta Kasada, Akihiko Kimura (Kyoto University) and Takuya Nagasaka (National Institute for Fusion Science)

In present study, the effects of neutron irradiation on the hardness of W-coated ODS steels were investigated. Vacuum plasma spraying (VPS) technique was employed to fabricate W layer on the surface of substrates. Substrate materials were K1-, K4-ODS ferritic steels and F82H steel, which are 3mm thick plates. Tungsten powder of 99.9% high purity was used for VPS process. The powders were sprayed by the plasma jet of argon and hydrogen mixture to the surface of substrates pre-annealed at 773K. VPS-W coated specimens were irradiated at 723K to 4.5, 9.6 dpa in the High Flux Isotope Reactor (HFIR), respectively. To investigate the effect of neutron irradiation on joint area, the hardness distributions were evaluated on the cross section using a micro Vickers hardness tester. Microstructure analysis revealed that W was successfully coated on each substrate by VPS process, in spite of mismatch of the CTE between these materials. The hardness of W was significantly varied from 260 to 480Hv because inhomogeneous cooling rate during the process. After irradiations, irradiation hardening was occurred in the VPS-W, while substrate materials did not.

5. CORROSION AND COMPTIBILITY

5.1 COMPATIBILITY OF MATERIALS EXPOSED TO ISOTHERMAL Pb-Li — **115**
B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory, USA)

Characterization of wrought and oxide dispersion strengthened (ODS) Fe-Cr alloys after exposure to Pb-Li in isothermal capsule experiments is in progress. Oxide precipitates appear to coarsen near the surface perhaps due to the loss of metallic substrate. To further investigate the performance of Al-rich coatings, a time series of exposures in Pb-Li is being conducted and results are compared to observations for uncoated Fe-Cr specimens. Measurement of the Al profile after 500h suggests that there is an initial Al loss from the coating on exposure. Dissimilar metal experiments with SiC capsules and Fe and SiC specimens have been completed and the mass change data suggests a large difference in behavior for the Fe specimens after 1000h at 700°C. Unfortunately, the SiC specimens chipped during loading/unloading resulting in unreliable mass change data.

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5.2 ADDITIONAL CHARACTERIZATION OF V-4Cr-4Ti EXPOSED TO FLOWING Li — 121
B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory)

In order to further understand the behavior of specimens exposed in the loop, microstructural characterization is being performed on the V-4Cr-4Ti tensile specimens that showed dynamic strain aging at 500°C. Initial results are presented. Also, further characterization on the dual layer MHD coatings is planned.

6. THEORY AND MODELING

6.1 First-Principles Investigation of the Influence of Alloying Elements on the Elastic and Mechanical Properties of Tungsten — 124
G. D. Samolyuk, Y. N. Osetskiy, and R. E. Stoller (Oak Ridge National Laboratory)

The properties of ½<111> screw dislocations in $W_{1-x}Tm_x$ alloys with different transition metal, Tm, concentrations within the local density approximation of density functional theory was investigated. The electronic structure of alloys was described within the virtual crystal approximation. It was demonstrated that alloying of W with transition metals from group VIII modifies the properties of screw dislocation similar to the effect of alloying with Re. Specifically, the alloying changes the dislocation core from symmetric to asymmetric and reduces the value of the Peierls barrier and Peierls stress. These modifications are caused by filling of d-electron states of W.

6.2 INFLUENCE OF CARBON ON HELIUM – DEFECT INTERACTIONS IN SINGLE CRYSTAL IRON FOLLOWING HELIUM IMPLANTATION: THERMAL HELIUM DESORPTION AND POSITRON LIFETIME MEASUREMENTS AND MODELING — 129
Xunxiang Hu (University of California, Berkeley), Donghua Xu, and Brian D. Wirth (University of Tennessee, Knoxville)

The thermal desorption spectra of helium from single crystalline iron samples was obtained as a function of implantation conditions using the Berkeley thermal helium desorption spectroscopy (THDS). The implantation conditions were He ion implantation energies of 10, 20, or 40 keV at a consistent dose level $1x10^{15}$ He/cm^2. Constant rate heating ramps were employed to thermally desorb the implanted helium. The present results, in which the single crystalline iron has a lower carbon content, indicate a significant effect of carbon on the helium desorption behavior, when compared to our previous measurements. The results

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indicate that major helium releasing peaks of the iron samples with higher purity show up at lower temperatures. A spatially dependent cluster dynamics model is utilized to reproduce the helium spectrum in BCC region. Meanwhile it provides an opportunity to optimize the basic thermodynamic and kinetic energy parameters of helium and small helium-vacancy clusters. The combination of the modeling and THDS measurement allows identification of possible mechanisms (e.g. shrinkage of He_3V_2) responsible for the measured helium desorption peaks. Furthermore, the model is also used to predict the depth dependence of helium and helium-defect clusters as a function of time and temperature during the THDS measurement. The samples with the implantation condition of 40 keV, $1x10^{15}$ He/cm^2 have been measured by positron annihilation spectroscopy, as an initial attempt to correlate a second experimental technique of the vacancy cluster population against the modeling predictions. Good agreement is obtained between the positron lifetime analysis and the modeling calculation. The recent work provided an overview of the self-consistency of the model predictions based on the assumptions made in the helium-point defect binding and interaction energies and diffusivities.

6.3 CLUSTER DYNAMICS MODELING OF NANO-SCALE DEFECT AGGLOMERATION IN THIN MOLYBDENUM FOILS BENCHMARKED BY IN-SITU ION IRRADIATION UNDER TEM — **143**

Donghua Xu and Brian D. Wirth (Department of Nuclear Engineering, University of Tennessee, Knoxville, TN 37996, USA)

In a recent coordinated effort, we have studied defect cluster dynamics in nanometer-thick molybdenum foils under 1 MeV Krypton ion irradiation at 80°C through both cluster dynamics computation and in-situ TEM experiments. By making direct one-to-one comparisons with experimentally observed cluster number density and size distribution as functions of foil thickness and irradiation dose and dose rate, significant validation and optimization of the model have been conducted in terms of both physical contents (damage production mode, identities of mobile defects) and parameterization (diffusivities of mobile defects). The optimized model exhibits excellent agreement with the current set of experiments both qualitatively and quantitatively, while further validation of the model and parameterization will continue to be pursued in the future with respect to Kr ion irradiation at varied temperatures and neutron irradiations on Mo.

7. IRRADIATION AND EXPERIEMNTAL METHODS AND ANALYSIS

No contributions this period.

8. IRRADIATION EXPERIMENTS AND TEST MATRICES

8.1 Design of the LOW Temperature DCT Experiment — **157**

M. A. Sokolov, J. McDuffee, D. Heatherly, (Oak Ridge National Laboratory)

An experiment has been designed, fabricated and assembled to irradiate disk compact tension (DCT) specimens of F82H in the flux trap of the High Flux Isotope Reactor (HFIR).

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Twelve DCT specimens with an outer diameter of 12.5 mm are exposed directly to HFIR coolant flow. The specimens are separated by perforated spacers to ensure cooling flow to the upper and lower surfaces of the specimens. Another experiment will use a perforated capsule to irradiate bend bar and tensile specimens to supplement this irradiation experiment, and provide the other specimen geometries for comparison with the DCT specimen data.

8.2 Reseach Plan and Status of High-Dose Rabbit irradiation Experiment for Silicon Carbide Composites — **164**

T. Nozawa, K. Ozawa, H. Tanigawa (Japan Atomic Energy Agency), Y. Katoh, L. L. Snead, and R. E. Stoller (Oak Ridge National Laboratory)

In order to examine the high-dose and high-temperature neutron irradiation effects on (1) mechanical properties of SiC, SiC/SiC composites and the fiber/matrix (F/M) interface, and (2) microstructural change of pyrolytic carbon (PyC) as the F/M interphase, new HFIR irradiation experiments were planned. Two types of rabbit capsules were designed: (1) composites' compact flexure rabbits for macro-strength evaluation and (2) mini-composites' rabbits for the F/M interfacial evaluation.