TABLE OF CONTENTS

1 VANADIUM ALLOYS

1.1 COMPRESSION TESTING OF V-4Cr-4Ti - M. B. Toloczko, R. M. Ermi, D. S. Gelles, R. J. Kurtz (Pacific Northwest National Laboratory), pp. 001-009. **1**

The NIFS-1 heat and heat 832665 of V-4Cr-4Ti, irradiated to 3.7 dpa at 425°C and tested in compression at temperatures of 25°C and ~420°C, were compared to compression tests at similar temperatures on unirradiated material. The yield strength increased by a factor of two, and the upper/lower yield point that was observed in the unirradiated material was not present in the irradiated material test traces. The strain hardening exponent of the irradiated material was 40-80% less than the unirradiated material. Transmission electron microscopy observations indicate the formation of a fine distribution of small defects and no dislocation channeling.

1.2 ADDITIONAL CHARACTERIZATION OF V-4Cr-4Ti AND MHD COATINGS EXPOSED TO FLOWING Li - B. A. Pint (Oak Ridge National Laboratory), pp. 010-013. **10**

Additional characterization is being performed on specimens exposed to flowing Li in a thermal gradient. These include 500°C tensile testing, measurement of changes in interstitial elements and characterization of the MHD coatings. To assist in the interpretation of these results, a group of specimens was annealed at 700°C for 2,350h in a quartz ampoule.

2 CERAMIC COMPOSITE MATERIALS

2.1 MECHANICAL, THERMAL, AND ELECTRICAL PROPERTIES OF NUCLEAR-GRADE SILICON CARBIDE COMPOSITES —R. Shinavski (Hyper-Therm HTC, Inc.), Y. Katoh and L. L. Snead (Oak Ridge National Laboratory), pp. 014-018. **14**

Nuclear grade SiC/SiC composites combine the attributes of high temperature mechanical strength and toughness with a relative dimensional stability under high neutron fluences that address the primary requirement of survivability in application as a flow channel insert. Unirradiated, through-thickness thermal conductivity has been identified as one property that is not suitable for a flow channel insert. However calculations indicate that through architecturally creating a structure of the nuclear grade SiC/SiC that the targeted low thermal conductivity can be achieved. A database of properties is being developed on a number of nuclear grade SiC/SiC composite. This data accumulation has not yet measured all relevant properties, but has set out a methodology for generating these properties and statistically reducing the properties to a property that can be used for component design.

2.2 MICROSTRUCTURAL EXAMINATION OF SiC_F/SiC WOVEN FIBER COMPOSITES – D. S. Gelles and G. E. Youngblood (Pacific Northwest National Laboratory), pp. 019-025. **19**

Electrical and thermal conduction in 2D-SiC_f/SiC composites exhibit lower than expected values in directions normal to the fiber weave plane. Transmission electron microscopy was used to carefully examine if micro-porosity possibly remaining at the impingement interface regions of the columnar SiC grains growing outwardly from adjacent SiC fiber surfaces could be partly responsible for the observed lower than expected transverse EC- and TC-values. Instead, in these regions no micro-porosity was observed; but rather a complete filling in of the vapor deposited SiC had occurred. The actual connectivity and amounts of the constituent phases (fibers, fiber coatings and CVI-SiC matrix) in each direction and the individual EC-values of the constituents govern the overall transverse and normal EC-values in 2D-SiC_f/SiC composite.

2.3 DEVELOPMENT AND EVALUATION OF SILICON CARBIDE JOINTS FOR APPLICATIONS IN RADIATION ENVIRONMENT —Y. Katoh (Oak Ridge National Laboratory), T. Hinoki, H.C. Jung, J.S. Park, and S. Konishi (Kyoto University), and M. Ferraris (Politecnico di Torino), pp. 026-032. **26**

Status of research and development of joining technology for silicon carbide-based ceramics and composites was surveyed and briefly summarized in terms of mechanical properties and anticipated stability in radiation environment. Several techniques which may be viable for joining silicon carbide-based materials for fusion and nuclear services were identified. Test methods appropriate for testing shear properties of silicon carbide joints, including testing of neutron-irradiated specimens, was studied and identified. Torsional shear of solid cross-section specimens with hourglass-shaped fillet sections was selected as the method to be employed in the irradiation study in US-Japan TITAN program. Several miniature test specimen geometries were examined both by finite element analysis and by experiment. Joint specimen with a <~5 mm-diameter joint section and square grip sections was identified acceptable for the initial irradiation campaign. A few issues associated with the use of small and non-ideal geometry specimens were identified and adequately addressed.

2.4 CONCENTRIC RING ON RING TEST FOR UNIRRADIATED AND IRRADIATED MINIATURE SIC SPECIMENS - S. Kondo, Y. Katoh, and L.L. Snead (Oak Ridge National Laboratory), pp. 033-040. **33**

The flexure strength of miniature disk specimens was evaluated for both the unirradiated and irradiated CVD SiC by equibiaxial flexural test, where a disk specimen was supported on a ring and centrally loaded with a smaller loading ring. The obtained mean flexural strength and Weibull modulus were σ_f = 352 MPa and m = 5.0 for unirradiated specimen, respectively. Both of them are relatively smaller than the typical values of uniaxial tests such as 4 point bend test previously reported. However, no stress magnification at the loading ring, which is often concerned in the biaxial tests for the disk specimens, was indicated by the observation of fracture patterns. Above the irradiation temperature of 1100˚C, the flexural strength is almost same as the unirradiated values or slightly decreased at 1500˚C in contrast to the strengthening observed previously at 300-800˚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 1100˚C. A substantially lower population of finer defect clusters such as loops, vacancy, and vacancy clusters may be attributed to the inhibition of the strengthening at the higher irradiation temperatures. Although, many factors, such as the effective flaw populations subject to stress, may influence the relation between equibiaxial and uniaxial strength of ceramics, the simple and rapid procedure of the ring-on-ring test may be favored for the study on irradiation effects on the mechanical properties of SiC.

3 FERRITIC/MARTENSITIC STEELS

3.1 A Comparison of Cavity Formation in Neutron Irradiated Nanostructured Ferritic Alloys and Tempered Martensitic Steels at High He/dpa Ratio – G.R. Odette, P. Miao, T. Yamamoto (Department of Mechanical Engineering, University of California Santa Barbara), D. J. Edwards, R Kurtz (Materials Science Division, Pacific Northwest National Laboratory) and H. Tanagawa (Japan Atomic Energy Agency), pp. 041-043. **41**

Microstructural evolutions in NFA under neutron-irradiation, at fusion relevant He/dpa ratios and dpa rates, were characterized using a novel in-situ ^{59}Ni(n,α) reaction He-implanter technique. MA957 was irradiated at 500°C in HFIR to a nominal 9 dpa and 380 appm He. A high number density of ≈ 1 He-bubbles were observed. Comparisons of these results to the cavity structures in TMS F82H, described in a companion report, provide additional evidence that TMS may be susceptible to both low (fast fracture) and high (creep rupture) temperature embrittlement as well as void swelling at fusion relevant He concentrations, while NFA are much more resistant to these degradation phenomena.

3.2 HELIUM EFFECTS ON MICROSTRUCTURAL EVOLUTION IN TEMPERED MARTENSITIC STEELS: IN SITU HELIUM IMPLANTER STUDIES IN HFIR - T. Yamamoto, G. R. Odette and P. Miao (Mechanical Engineering Department and Materials Department, University of California, Santa Barbara), D. J. Edwards and R. J. Kurtz (Pacific Northwest National Laboratory), pp. 044-052. **44**

Microstructural evolutions in TMS under neutron-irradiation, at fusion relevant He/dpa ratios and dpa rates, were characterized using a novel in-situ ^{59}Ni(n,α) reaction He-implanter technique. F82H-mod3 was irradiated at 500°C in HFIR to a nominal 9 dpa and 190 or 380 appm He in both in the as-tempered (AT) and 20% cold-worked (CW) conditions. In all cases, a high number density of 1-2 nm He-bubbles were observed, along with a smaller number of larger faceted ≈ 10 nm cavities, which are likely voids. The He bubbles form preferentially on dislocations and various interfaces, including grain boundaries and at precipitates. A slightly larger number of smaller He bubbles were observed in the CW condition. The lower He/dpa ratio produced slightly smaller and fewer He bubbles. Estimates of the He content of the bubbles, assuming equilibrium capillary pressures, were in good agreement with the nominal implanted levels. Comparisons of these results to the cavity structures in nano-structured ferritic alloy (NFA) MA957, described in a companion report [1], provide additional evidence that TMS may be susceptible to both low (fast fracture) and high (creep rupture) temperature embrittlement as well as void swelling at fusion relevant He concentrations, while NFA are much more resistant to these degradation phenomena.

3.3 THE MICROSTRUCTURAL STABILITY OF A RUPTURED THERMAL CREEP SPECIMEN OF MA957 - D.T. Hoelzer, J.P. Shingledecker, R.L. Klueh, M.K. Miller, and J. Bentley (Oak Ridge National Laboratory), pp. 053-061. **53**

A set of thermal creep tests were conducted on as-received MA957 starting in August 2003 shortly after discovering a high number density of Ti-, Y-, and O-enriched nanoclusters that were similar to those that had been discovered in 12YWT by APT in 1999. Four of the creep tests that were conducted at temperatures between 875ºC and 925ºC with 70 or 100 MPa loads ended with failure of the specimens before reaching 2000 h. On the other hand, two of the creep tests were conducted at lower temperatures of 800ºC (100 MPa) and 825ºC (70 MPa) and these lasted a considerably longer time. However, the specimen that was tested at 800ºC and 100 MPa recently failed after 38,555 h while the other specimen that was tested at 825ºC and 70 MPa is still in progress with more than 43,000 h logged to date. The microstructure of the recently failed creep specimen was investigated using optical microscopy and TEM, including Energy-Filtered TEM (EFTEM). The optical microscopy and TEM results showed that extensive porosity formed throughout the microstructure during the creep test. However, the significant discovery obtained by EFTEM revealed that the nanoclusters experienced no significant change in size, indicating that they are extremely stable at 800ºC (and 100 MPa) for very long periods of time.

3.4 ON THE STATIC AND CREEP STRENGTH OF MA957 FROM ROOM TEMPERATURE TO 1000ºC - M.C. Salston and G.R. Odette (Department of Mechanical Engineering, University of California, Santa Barbara), pp. 062-066. **62**

Static tensile and creep tests were carried out on as-extruded (AE) MA957 from room temperature to 1000ºC. Comparison of these results to data taken from the literature show the yield stress (σ_y) and ultimate tensile strength of MA957 are generally higher than for 9 Cr tempered martensitic steels (TMS), like Eurofer97. However, the static tensile strength of MA957 varies by up to a factor of 2, or more, depending on the post extrusion thermal mechanical heat treatment (TMT) and specimen orientation with respect to the extrusion direction. The corresponding creep strength varies by up to a factor of ≈ 10. The AE and TMT variants of MA957 were fit to a threshold stress (σ_{tr}) model. The $\sigma_{tr}(T)$ for the AE MA957 is $\geq 0.4\sigma_y(T)$ up to 800°C and decreases at higher temperatures, approaching 0 at 1000°C.

3.5 COMPATIBILITY OF MATERIALS EXPOSED TO ISOTHERMAL Pb-Li – B. A. Pint (Oak Ridge National Laboratory, USA), pp. 067-071. **67**

A series of six Pb-Li capsule experiments were conducted at 700° and 800°C for 1,000h using commercial purity Pb-17Li. The use of commercial Pb-Li with a higher O content did not reduce the amount of dissolution observed for type 316 stainless steel (316SS) at 700°C. The amount of dissolution for Fe-9Cr-2W (T92) was similar to 316SS at this temperature. However, when an Al-rich diffusion coating was applied to T92, the specimen mass loss was greatly reduced. The aluminized T92 specimen as well as a FeCrAl specimen formed $LiAlO_2$ on the surface. Exposures of FeCrAl and NiAl specimens at 700° and 800°C, pre-oxidized to form α-Al_2O_3, confirmed the prior observation that alumina transforms to $LiAlO_2$ during exposure to PbLi.

4 COPPER ALLOYS

No contributions this period

5 REFRACTORY METALS AND ALLOYS

No contributions this period

6 AUSTENITIC STAINLESS STEELS

6.1 SEVERE EMBRITTLEMENT OF NEUTRON IRRADIATED AUSTENITIC STEELS ARISING FROM HIGH VOID SWELLING – V. S. Neustroev (Research Institute of Atomic Reactors, Dimitrovgrad, Russia) and F. A. Garner (Pacific Northwest National Laboratory, Richland WA USA), pp. 072-079. **72**

Data are presented from BOR-60 irradiations showing that significant radiation-induced swelling causes severe embrittlement in austenitic stainless steels, reducing the service life of structural components and introducing limitations on low temperature handling especially. It is shown that the degradation is actually a form of quasi-embrittlement arising from intense flow localized deformation with high levels of localized ductility involving micropore coalescence and void-to-void cracking. Voids initially serve as hardening components whose effect is overwhelmed by the void-induced reduction in shear and Young's moduli at high swelling levels. Thus the alloy appears to soften even as the ductility plunges toward zero on a macroscopic level although a large amount of deformation occurs microscopically at the failure site. Thus the failure is better characterized as "quasi-embrittlement" which is a suppression of uniform deformation. This case should be differentiated from that of real embrittlement which involves the complete suppression of the material's capability for plastic deformation.

6.2 UNUSUAL ENHANCEMENT OF DUCTILITY OBSERVED DURING EVOLUTION OF A DEFORMATION WAVE IN 12Cr18Ni10Ti STAINLESS STEEL IRRADIATED IN BN-350 – M. N. Gusev, N. S. Silniagina, I. S. Osipov, O. P. Maksimkin (Institute of Nuclear Physics, Almaty, Kazakhstan) and F. A. Garner (Pacific Northwest National Laboratory, Richland WA USA), pp. 080-087. **80**

Whereas most previous irradiation studies conducted at lower neutron exposures in the range 100–400°C have consistently produced strengthening and strongly reduced ductility in stainless steels, it now appears possible that higher exposures may lead to a reversal in ductility loss for some steels. A new radiation-induced phenomenon has been observed in 12Cr18Ni10Ti stainless steel irradiated to 26-55 dpa. It involves "a moving wave of plastic deformation" at 20–60°C that produces "anomalously" high values of engineering ductility, especially when compared to deformation occurring at lower neutron exposures. Due to the concentrated deformation occurring at the wave front, the wave moves much faster than the mechanically applied strain rate. However, when strained at 120°C the moving wave is not observed, indicating that the phenomenon operates at lower test temperatures. Using the technique of digital optical extensometry the "true stress–true strain" curves were obtained. It appears that the moving wave of plastic deformation occurs as a result of an increase in the intensity of strain hardening, $d\sigma/d\varepsilon(\varepsilon)$. The increase in strain hardening is thought to arise from an irradiation-induced increase in the propensity of the $\gamma\rightarrow\alpha$ martensitic transformation.

7 MHD INSULATORS, COATINGS, INSULATING CERAMICS, AND OPTICAL MATERIALS

See contribution 1.2

8 BREEDING MATERIALS

No contributions this period

9 RADIATION EFFECTS, MECHANISTIC STUDIES, AND EXPERIMENTAL METHODS

See ALSO 2.3 and 2.4 related to Test Methods

9.1 MACROSCOPIC DEFORMATION MODES AND STRESS PARAMETERS IN METALLIC MATERIALS AFTER LOW TEMPERATURE IRRADIATION – T. S. Byun and K. Farrell (Oak Ridge National Laboratory) and M. Li (Argonne National Laboratory), pp. 088-108. **88**

Macroscopic deformation modes, elastic, uniform plastic, and unstable plastic deformation modes, are mapped in tensile true stress-dose space for more than two dozen metallic materials consisting of 13 bcc, 11 fcc, and 2 hcp metals irradiated at low temperatures (≤ 200°C). The boundaries between different deformation regions are set by the yield stress (YS), plastic instability stress (PIS), and true fracture stress (FS) versus dose curves. The annealed fcc metals display large uniform plasticity regions, while unstable deformation regions are dominant in the harder bcc and hcp metals. The PIS values for all materials are independent of dose except for the precipitation-hardened IN718 alloy where the irradiation-induced phase change reduces its PIS. In the bcc materials for high temperature application, such as 9Cr ferritic/martensitic steels, sintered molybdenum, vanadium, and tantalum, the radiation-induced embrittlement is characterized in terms of FS decreasing with dose at relatively high doses. The FS is nearly dose-independent below the critical dose for the embrittlement. It is concluded that the tensile stress-based deformation mode maps effectively integrate mechanical property information and characterize differences in radiation effects between crystalline structures or material groups. Also, the analysis results indicate that the low temperature irradiation does not significantly change the strain-hardening rate of metallic materials. Such a dose independence in strain hardening behavior results in strong linear relationships between the true stress parameters.

9.2 DISSOCIATION OF MIGRATING PARTICLES FROM TRAPS WITH A LONG-RANGE INTERACTION FIELD.- A.V. Barashev (The University of Liverpool), S.I. Golubov (Oak Ridge National Laboratory, University of Tennessee), Yu.N. Osetsky and R.E. Stoller (Oak Ridge National Laboratory), pp. 109-121. **109**

The main results can be summarized as follows:

1. An equation for the mean dissociation time of a migrating particle from a trap has been derived. It is independent of the saddle point energy profile within the well.
2. Generally, the distribution of dissociation times deviates from an exponential function, especially in the regime of small dissociation times. The probability function at the mean time may differ significantly from $1-e^{-1}$, which is for a random process. The effect depends on the well shape (is stronger for shallower and/or wider wells) and on the saddle point energy profile within the well.
3. The exchange frequency for diffusing particles between spatially separated wells is generally many orders of magnitude smaller than the frequency for dissociation from the well, and this is due to correlated recapture of diffusing particles by the same well.

Note also that, in general, the information on the mean dissociation time alone and, hence, the effective binding energy associated with it, is not sufficient to characterise the process completely and the probability distribution function of dissociation times has to be taken into consideration. Work is currently in progress to investigate the description of complexes with a non-exponential distribution of dissociation times by chemical reaction rate equations.

9.3 ATOMISTIC STUDIES OF PROPERTIES OF HELIUM IN BCC IRON USING THE NEW HE–FE POTENTIAL—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), pp. 122-126. **122**

In fusion applications, helium caused by transmutation plays an important role in the response of RAFM steels to neutron radiation damage. The growth, migration and coalescence behavior of helium bubbles is very sensitive to the properties of individual He interstitials and helium-vacancy clusters. We have performed atomistic simulations using a new 3-body Fe–He inter-atomic potential combined with the Ackland iron potential. With the ORNL potential, interstitial helium is very mobile and coalesces together to form interstitial clusters. The interstitial clusters show lower binding energy than with the Wilson potential, in agreement with the DFT calculations of CC Fu. If the He cluster is sufficiently large the cluster can push out an Fe interstitial, creating a Frenkel pair. The resulting helium-vacancy cluster is not mobile. The ejected SIA is mobile, but is weakly trapped by the He-V cluster. If more helium atoms join the He-V cluster, more Fe interstitials can be pushed out, and they combine to form an interstitial dislocation loop. Such loops have been observed in other experiments. The reverse process is also studied. Multiple helium atoms can be trapped in a single vacancy, and if there are few enough, the vacancy can recombine with an Fe interstitial to create a helium interstitial cluster.

10 DOSIMETRY, DAMAGE PARAMETERS, AND ACTIVATION CALCULATIONS
No contributions this period

11 MATERIALS ENGINEERING AND DESIGN REQUIREMENTS
No contributions this period

12 IRRADIATION FACILITIES AND TEST MATRICES
No contributions this period