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Calculations were performed to estimate damage and transmutation rates in vanadium irradiated in the ATR (Advanced Test Reactor) located in Idaho. The main focuses of the study are to evaluate the transmutation of vanadium to chromium and to explore ways to design the irradiation experiment to avoid excessive transmutation. It was found that the A-hole of ATR produces damage rate of ~ 15 -30 dpa/full power year with a transmutation rate of $\sim 0.2\%$ /dpa of vanadium to chromium. A thermal neutron filter can be incorporated into the design to reduce the vanadium-to-chromium transmutation rate to low levels. A filter 1-2-mm thick of gadolinium or hafnium can be used.

- 8.10 ELASTIC STABILITY OF HIGH DOSE NEUTRON IRRADIATED SPINEL –
Z. Li and S.-K. Chan (Argonne National Laboratory), F. A. Garner (Pacific Northwest
Laboratory), and R. C. Bradt (University of Nevada-Reno). 362

Elastic constants (C_{11} , C_{12} , and C_{44}) of spinel ($MgAl_2O_4$) single crystals irradiated to very high neutron fluences have been measured by an ultrasonic technique. Although results of a neutron diffraction study show that cation occupation sites are significantly changed in the irradiated samples, no measurable differences occurred in their elastic properties. In order to understand such behavior, the elastic properties of a variety of materials with either normal or inverse spinel structures were studied. The cation valence and cation distribution appear to have little influence on the elastic properties of spinel materials.

- 8.11 OPTICAL ABSORPTION AND LUMINESCENCE IN NEUTRON-IRRADIATED,
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B. L. Bennett, B. Sundlof, And W. P. Unruh (Los Alamos National Laboratory). 379

ITER plasma performance will be monitored by fiber optics cables capable of transmitting light of various wavelengths in the ultraviolet, visible, and near infrared regime. The fibers must retain their optical integrity under extreme conditions of elevated temperature and mixed neutron and γ -ray exposure. Previous research has shown that fibers composed of pure silica cores and fluorine-doped cladding might be appropriate candidates for this application. Optical absorption and emission on commercially-available silica fibers containing both low (<1 ppm) and high (600-800 ppm) OH concentrations, that had been irradiated at the Los Alamos Spallation Radiation Effects Facility (LASREF) to a fluence of 10^{23} n-m², were measured in the wavelength interval 200-800 nm. Generally the low-OH fiber performance was superior, although neither fiber exhibited the optimum optical integrity required for ITER diagnostic applications. Both unirradiated fibers showed good transmissivity in the region of 400-800 nm; however, following irradiation each exhibited strong absorption over the entire wavelength region except for a small interval around 400 to 500 nm. Attempts to thermally anneal or photobleach the radiation-induced damage were only partially successful. In addition to the poor transmission properties of the irradiated fibers in the 200-800 nm region, there was intrinsic luminescence near 460 nm that occurred during exposure to *continuous* x irradiation at room temperature. This emission, if induced by neutrons and γ -rays of the ITER environment, would interfere with optical diagnostic measurements.

- 2.2 NEUTRON FLUX SPECTRA AND RADIATION DAMAGE PARAMETERS FOR THE RUSSIAN BOR-60 AND SM-2 REACTORS – A. V. Karasiov (D. V. Efremov Scientific Research Institute of Electrophysical Apparatus, St. Petersburg, Russia) and L. R. Greenwood (Pacific Northwest Laboratory). 21

Neutron fluence and spectral information and calculated radiation damage parameters are presented for the BOR-60 (Fast Experimental Reactor - 60 MW) and SM-2 reactors in Russia. Their neutron exposure characteristics are comparable with those of the Experimental Breeder Reactor (EBR-II), the Fast Flux Test Facility (FFTF), and the High Flux Isotope Reactor (HFIR) in the United States.

- 2.3 NEUTRON DOSIMETRY, DAMAGE CALCULATIONS, AND HELIUM MEASUREMENTS FOR THE HFIR-MFE-60J-1 AND MFE-330J-1 SPECTRAL TAILORING EXPERIMENTS – L. R. Greenwood (Pacific Northwest Laboratory), C. A. Baldwin (Oak Ridge National Laboratory), and B. M. Oliver (Rockwell International). 28

Neutron fluence measurements and radiation damage calculations are reported for the joint U.S.-Japanese MFE-60J-1 and MFE-330J-1 experiments in the hafnium-lined removable beryllium (RB*) position of the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). These experiments were continuations of the ORR-6J and 7J irradiations performed in the Oak Ridge Research Reactor (ORR). The combination of irradiations was designed to tailor the neutron spectrum in order to achieve fusion reactor helium/dpa levels in stainless steel. These experiments produced maximum helium (appm)/dpa (displacement per atom) levels of 10.2 at 18.5 dpa for the ORR-6J and HFIR-MFE-60J-1 combination and 11.8 at 19.0 dpa for the ORR-7J and HFIR-MFE-330J-1 combination. A helium measurement in one JPCA sample was in good agreement with helium calculations.

- 2.4 PRELIMINARY REPORT ON THE IRRADIATION CONDITIONS OF THE HFIR JP-23 EXPERIMENT – A. M. Ermi (Westinghouse Hanford Company) and D. S. Gelles (Pacific Northwest Laboratory). 35

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- 3.0 MATERIALS ENGINEERING AND DESIGN REQUIREMENTS. 51

NO CONTRIBUTIONS

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- 4.1 CORRELATION BETWEEN SHEAR PUNCH AND TENSILE DATA FOR NEUTRON-IRRADIATED ALUMINUM ALLOYS – M. L. Hamilton and D. J. Edwards (Pacific Northwest Laboratory), M. B. Toloczko and G. E. Lucas (University of California-Santa Barbara), W. F. Sommer and M. J. Borden (Los Alamos National Laboratory), and J. F. Dunlap and J. F. Stubbins (University of Illinois). 55

Tensile specimens and TEM disks of two aluminum alloys in two tempers were irradiated at 90-120°C with neutrons from spallation reactions in the Los Alamos Spallation Radiation Effects Facility (LASREF) at the Los Alamos Meson Physics Facility (LAMPF). The materials were exposed to a fluence of $3.4\text{--}4 \times 10^{20}$ n/cm². This work was part of a study to determine the potential for using aluminum alloys as structural components in accelerators used to produce tritium. Shear punch tests and tensile tests were performed at room temperature and at 100°C, a temperature characteristic of the operating temperature in the application of interest. Shear punch and tensile data from unirradiated specimens were used to develop a correlation between shear punch and tensile strengths. Using the shear punch data from the neutron irradiated TEM disks, the correlation predicted the tensile strength of the

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5.1 COMPARISON OF DEFECT CLUSTER ACCUMULATION AND PATTERN FORMATION IN IRRADIATED COPPER AND NICKEL – S. J. Zinkle and L. L. Snead (Oak Ridge National Laboratory), B. N. Singh (Risø National Laboratory), and D. J. Edwards (Pacific Northwest Laboratory). 69

Transmission electron microscopy was used to examine the density and spatial distribution of defect clusters produced in copper and nickel as the result of fission neutron irradiation to damage levels of 0.01 to 0.25 displacements per atom (dpa) at irradiation temperatures between 50 and 230°C (0.24 to 0.37 T_M in Cu). A high density of small stacking fault tetrahedra (SFT) and dislocation loops was observed in both materials, and a moderate density of small voids was observed in the copper specimens irradiated at 230°C. The visible defect cluster density in both materials approached a saturation value at doses >0.1 dpa. The visible defect cluster density in nickel was a factor of 5 to 10 lower than that in copper at all damage levels. The defect clusters in Ni organized into {001} walls at damage levels >0.1 dpa, whereas defect cluster alignment was not observed in copper. A comparison with published results in the literature indicates that defect cluster wall formation occurs in nickel irradiated at 0.2 to 0.4 T_M in a wide variety of irradiation spectra. Defect cluster wall formation apparently only occurs in copper during low temperature irradiation with electrons and light ions. These results are discussed in terms of the thermal spike model for energetic displacement cascades.

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6.1.1 EFFECT OF INTERNAL HYDROGEN ON THE MIXED-MODE I/III FRACTURE TOUGHNESS OF A FERRITIC/MARTENSITIC STAINLESS STEEL - H. Li (Associated Western Universities-Northwest Division), R. H. Jones (Pacific Northwest Laboratory), J. P. Hirth (Washington State University), and D. S. Gelles (Pacific Northwest Laboratory). 85

The effects of H on the mixed-mode I/III critical J_{TQ} and tearing moduli (dJ/da) were examined for a ferritic/martensitic stainless steel at ambient temperature. A H content of 4 ppm (wt) was attained after charging in a H_2 gas chamber (138 MPa) at 300°C for 2 weeks. Results showed that H decreased J_{TQ} and dJ/da values compared to steel tested without H. However, the presence of H did not change the dependence of J_{TQ} and dJ/da values on crack angle. Both J_{TQ} and dJK/da exhibited the highest relative values. The minimum values of both J_{TQ} and dJ/da occurred at a crack angle between 35 and 55° [$P_{iii} + P_i$]/($P_{iii} + P_i$) = 0.4 and 0.6]. A mechanism of the combined effect of H and mixed-mode on J_{TQ} and dJ/da is discussed.

6.1.2 DEPENDENCE OF MODE I AND MIXED MODE I/III FRACTURE TOUGHNESS ON TEMPERATURE FOR A FERRITIC/MARTENSITIC STAINLESS STEEL H. Li (Associated Western Universities-Northwest Division), R. H. Jones (Pacific Northwest Laboratory), J. P. Hirth (Washington State University), and D. S. Gelles (Pacific Northwest Laboratory). 99

Mode I and mixed mode I/III fracture toughnesses were investigated in the range of -95°C to 25°C for a F82-H steel heat-treated in the following way; 1000°C/20 h/air-cooled (AC), 1100°C/7 min/AC, and 700°C/2 h/AC. Mode I fracture toughness (J_{IC}) was determined with standard compact tension (CT) specimens, and mixed-mode I/III fracture toughness (J_{MC}) was determined with modified CT specimens, which resulted in 0.41 ratio of

$P_{III}/(P_{II} + P_I)$. The F82-H was very tough at room temperature (RT), giving a J_{IC} value of about 284 kJ/m². Mixed-mode I/III loading dramatically lowered fracture toughness. The J_{MC} value was only 150 kJ/m² at RT. J_{IC} values exhibited a strong temperature dependence and decreased rapidly with decrease of temperature. J_{IC} at -90°C was only 30 kJ/m². On the other hand, J_{MC} values depended much more weakly on temperature. J_{MC} at -95°C was 50kJ/m², about 70% higher than J_{IC} value at -90°C. At RT the mode I specimens fractured by microvoid coalescence. Mixed mode specimens also fractured by microvoid coalescence, but tortuosity and void size on fracture surfaces were significantly less than those in mode I specimens. Interestingly, at -90°C, a crack in a mode I specimen initiated and grew by quasi-cleavage fracture; but a crack in mixed mode specimen initiated and propagated a short distance (0.5 mm) by a mixture of intergranular fracture and ductile tearing. Then the crack turned to mode I and fractured by quasi-cleavage failure. Our results indicate that crack tip plasticity was increased by mixed mode loading, and suggest that at low temperature, mode I fracture toughness is the critical design parameter, but at temperatures above RT, especially concerning fatigue and creep-fatigue crack growth rate, a mixed mode loading may be more harmful than a mode I loading for this steel because a mixed mode loading results in lower fracture toughness and higher crack tip plasticity (or dislocation activity).

6.1.3 EMBRITTLEMENT OF Cr-Mo STEELS AFTER LOW FLUENCE IRRADIATION IN HFIR – R. L. Klueh and D. J. Alexander. 110

Subsize Charpy impact specimens of 9Cr-1MoVNb (modified 9Cr-1Mo) and 12Cr-1MoVW (Sandvik HT9) steels and 12Cr-1MoVW with 2% Ni (12Cr-1MoVW-2Ni) were irradiated in the High Flux Isotope Reactor (HFIR) at 300 and 400°C to damage levels up to 2.5 dpa. The objective was to study the effect of the simultaneous formation of displacement damage and transmutation helium on impact toughness. Displacement damage was produced by fast neutrons, and helium was formed by the reaction of ⁵⁸Ni with thermal neutrons in the mixed-neutron spectrum of HFIR. Despite the low fluence relative to previous irradiations of these steels, significant increases in the ductile-brittle transition temperature (DBTT) occurred. The 12Cr-1MoVW-2Ni steel irradiated at 400°C had the largest increase in DBTT and displayed indications of intergranular fracture. A mechanism is proposed to explain how helium can affect the fracture behavior of this latter steel in the present tests, and how it affected all three steels in previous experiments, where the steels were irradiated to higher fluences.

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6.2.1 INITIAL TENSILE TEST RESULTS FROM J316 STAINLESS STEEL IRRADIATED IN THE HFIR SPECTRALLY TAILORED EXPERIMENT -- J. E. Pawel, M. L. Grossbeck, A. F. Rowcliffe (Oak Ridge National Laboratory) and K. Shiba (Japan Atomic Energy Research Institute). 125

The HFIR-MFE-RB* experiments are designed for irradiation in the removable beryllium (RB*) positions of the High Flux Isotope Reactor. A hafnium shield surrounds the capsules in order to reduce the thermal neutron flux and achieve a He/dpa level near that expected in a fusion reactor. The J316 austenitic alloy specimens irradiated in this experiment were in the solution annealed (SA) and 20% cold-worked (CW) condition. The specimens were irradiated at 60 and 330°C to a total of 19 dpa (11 appm He/dpa). For both irradiation temperatures, there was no significant difference between the strength properties of the CW J316 following irradiation to 7 dpa or 19 dpa. The strength properties saturate at a fluence less than 7 dpa. The same is true for the SA J316 irradiated at 60°C. However, at 330°C, there is a small but significant further increase in yield stress between 7 and 19 dpa. There is a marked difference in deformation behavior seen after irradiation at 60°C and 330°C. After irradiation to 19 dpa at 60°C, J316 maintains a uniform elongation greater than 20% while the uniform elongation of the 330°C material is less than 0.5%. The yield strength of the cold-worked material remains higher than that of the solution

annealed material at both 7 and 19 dpa. The severe reduction in uniform elongation seen at 330°C is a synergistic effect of both the irradiation temperature and the test temperature.

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6.3.1 CHEMICAL AND MECHANICAL INTERACTIONS OF INTERSTITIALS IN V-5%Cr-5%Ti – J. H. DeVan, J. R. DiStefano, J. W. Hendricks, and C. E. Matos (Oak Ridge National Laboratory). 137

Gas-metal reaction studies of V-5Cr-5Ti were conducted to determine the kinetics of reactions with H₂ and O₂, respectively, at 450-500°C. Reaction rates were determined through weight change measurements and chemical analyses, and effects on mechanical properties were evaluated by room temperature tensile tests. Exposures to hydrogen at 450°C and 0.1 torr pressure resulted in a significant loss in room temperature ductility in the case of alloys that has been annealed at 1125°C but not in the case of alloys annealed at 1050°C. Adding oxygen at 500°C at concentrations as low as 200 ppm seriously embrittled V-5Cr-5Ti specimens when the specimens were held for 100 h in vacuum at 500°C. A subsequent heat treatment in vacuum at 950°C restored the ductility. Exposure to air at 400°C and a subsequent vacuum heat treatment at 500°C caused ductility decreases similar to those observed after the small oxygen additions, and ductility again was restored by a 950°C vacuum anneal. However, similar heat treatments following air exposures at 450 and 500°C, respectively, resulted in ductility losses that were not recovered by the 950°C anneal. The latter exposures also resulted in the formation of thin oxide films.

6.3.2 FATIGUE BEHAVIOR OF UNIRRADIATED V-5Cr-5Ti – B. G. Gieseke, C. O. Stevens, and M. L. Grossbeck (Oak Ridge National Laboratory). 142

The results of in-vacuum low cycle fatigue tests are presented for unirradiated V-5Cr-5Ti tested at room temperature (25), 250, and 400°C. A comparison of the fatigue data generated in rough and high vacuums shows that a pronounced environmental degradation of the fatigue properties exists in this alloy at room temperature. Fatigue life was reduced by as much as 84%. Cyclic stress range data and SEM observations suggest that this reduction is due to a combination of increases in rates of crack initiation and subsequent growth. The relative contribution of each difference is dependent upon the strain range.

In high vacuum, the fatigue results also show a trend of increasing cyclic life with increasing temperature between 25 and 400°C. From the limited data available, life at 250°C averages 1.7 times that at 25°C, and at 400°C, life averages 3.2 times that at room temperature. Like the environmental effects at 25°C, the effect of temperature seems to be a function of strain range at each temperature.

The total strain range and cycles to failure were correlated using a power law relationship and compared to 20% cold-worked 316 stainless steel and several vanadium-base alloys. The results suggest that V-5Cr-5Ti has better resistance to fatigue than 316-SS in the temperature range of 25 to 400°C. At 400°C, the data also show that V-5Cr-5Ti outperforms Vanstar alloys 7 and 8 over the entire range of strains investigated. Furthermore, the fatigue properties of the V-5Cr-5Ti alloy compare favorably to V-15Cr-5Ti (at 25°C) and Vanstar 9 (at 400°C) at strains greater than 1%. At lower strains, the lower fatigue resistance of V-5Cr-5Ti is attributed to the higher strengths of the V-15Cr-5Ti and Vanstar 9 alloys.

6.3.3 WELDING DEVELOPMENT FOR V-Cr-Ti ALLOYS – J. F. King, G. M. Goodwin, and D. J. Alexander (Oak Ridge National Laboratory). 152

The subsize charpy test results for electron beam weld metal from the V-5Cr-5Ti alloy have shown significant improvement in charpy fracture energy compared to both GTA weld metal and the base metal itself. These results are preliminary, however, and additional confirmation testing and analysis will be required to explain this improvement in properties.

- 6.3.4 DISLOCATION DEVELOPMENT IN V-5Cr-5Ti AND PURE VANADIUM –
D. S. Gelles (Pacific Northwest Laboratory) and M. L. Grossbeck (Oak Ridge
National Laboratory). 156

Microstructural examinations have been performed on deformed tensile specimens of V-5Cr-5Ti and pure vanadium in order to explain notch sensitivity noted in the candidate alloy V-5Cr-5Ti. SS-3 tensile specimens have been prepared, stress relieved and deformed to 5% strain. The resulting deformation structures have been examined by transmission electron microscopy. It is found that 5% deformation in V-5Cr-5Ti produces a higher dislocation density consisting of long straight dislocations, typical of Stage II, and many small loops, whereas in pure vanadium, the dislocation arrangements are more complex, typical of Stage III, and the small loops are at a lower density.

These results are interpreted in light of the tendency for enhanced notch sensitivity found in V-5Cr-5Ti.

- 6.3.5 EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE AND FRACTURE
TOUGHNESS OF A V-5Cr-5Ti ALLOY – H. Li (Associated Western Universities-
Northwest Division), M. L. Hamilton and R. H. Jones (Pacific Northwest Laboratory). . . 165

Fracture toughness and impact tests were performed on a V-5Cr-5Ti alloy. Specimens annealed at 1125°C for 1 h and furnace cooled in a vacuum of 1.33×10^{-5} Pa were brittle at room temperature (RT) and experienced a mixture of intergranular and cleavage fracture. Fracture toughness (J_{IQ}) at RT was 52 kJ/m² and the impact fracture energy (IFE) was 6J. The IFE at -100°C was only 1 J. While specimens exhibited high fracture toughness at 100°C (J_{IQ} is 485 kJ/m²), fracture was a mixture of dimple and intergranular failure, with intergranular fracture making up 40% of the total fracture surface. The ductile to brittle transition temperature (DBTT) was estimated to be about 20°C. When some specimens were given an additional annealing at 890°C for 24 h, they became very ductile at RT and fractured by microvoid coalescence. The J_{IQ} value increased from 52 kJ/m² to ~1100 kJ/m². The impact test failed to fracture specimens at RT due to a large amount of plastic deformation. The IFE at -115°C was 4J, four times as much as when annealed only at 1125°C. The specimens became brittle at -50°C and fractured by cleavage, giving a J_{IQ} value of 50 kJ/m². The DBTT was estimated to be -40°C. Analysis of Auger electron microscopy showed significant sulfur segregation (6 at. %) on grain boundaries in the specimens annealed only at 1125°C, but only 0.9 at. % on grain boundaries if the additional annealing at 890°C was given. Moreover, significantly more second phase particles were found in the specimens annealed at 1125°C plus 890°C. The possible mechanism by which heat treatment affects fracture toughness is discussed.

- 6.3.6 FABRICATION OF 500-kg HEAT OF V-4Cr-4Ti – H. M. Chung, H.-C. Tsai, and
D. L. Smith (Argonne National Laboratory) and R. Peterson, C. Curtis, C. Wojcik,
and R. Kinney (Teledyne Wah Chang Albany). 178

A 500-kg heat of V-4Cr-4Ti, and alloy identified previously as the primary vanadium-based candidate alloy for application in fusion reactor structural components, has been produced. The ingot was produced by electron-beam melting using screened high-quality raw materials of vanadium and titanium. Several ~63.5-mm-thick bars were extruded from the ingot, and plates and sheets of various thicknesses ranging from 0.51 to 12.7 mm were fabricated successfully from the extruded bars. The chemical composition of the ingot and the secondary fabrication procedures, specified on the basis of experience and knowledge gained from fabrication, testing, and microstructural examination of a laboratory-scale heat, were found to be satisfactory. Charpy-impact tests showed that mechanical properties of the large-scale heat are as good as those of the laboratory-scale heat. This demonstrates a method of reliable fabrication of industrial-scale heats of V-4Cr-4Ti that exhibit excellent properties.

- 6.3.7 **IMPACT PROPERTIES OF 500-kg HEAT OF V-4Cr-4Ti** – H. M. Chung, L. Nowicki, J. Gazda, and D. L. Smith (Argonne National Laboratory). 183
- A 500-kg heat of V-4Cr-4Ti, an alloy identified previously as the primary vanadium-based candidate alloy for application as fusion reactor structural components, has been produced successfully. Impact tests were conducted at -196 to 150°C on 1/3-size Charpy specimens of the scale-up heat after final annealing for 1 h at 950, 1000, and 1050°C. The material remained ductile at all test temperatures, and the ductile-brittle transition temperature (DBTT) was lower than -200°C. The upper-shelf energy of the production-scale heat was similar to that of the laboratory-scale (≈ 15 -kg) heat. Effect of annealing temperature was not significant; however, annealing at 1000°C for 1 h produced impact properties slightly better than those from other annealing treatments. Effect of notch geometry was also investigated on the heat. Under otherwise similar conditions, DBTT increased $\approx 30^\circ\text{C}$ when the notch angle was reduced from 45° (root radius 0.25 mm) to 30° (root radius 0.08 mm).
- 6.3.8 **HARDNESS RECOVERY OF 85% COLD-WORKED V-Ti AND V-Cr-Ti ALLOYS UPON ANNEALING AT 180°C TO 1200°C** – B. A. Loomis, L. J. Nowicki, and D. L. Smith (Argonne National Laboratory). 187
- Annealing of 85% cold-worked unalloyed V and V-(1-18)Ti alloys for 1 hr at 180°C to 1200°C results in hardness maxima at 180-250°C, 420-600°C, and 1050-1200°C and in hardness minima at 280-360°C and, depending on Ti concentration in the alloy, at 850-1050°C. Annealing of 85% cold-worked V-(4-15)Cr-(3-6)Ti alloys for 1 hr at 180-1200°C results in hardness maxima at 180-250°C, 420-800°C and 1050-1200°C, and in hardness minima at 280-360°C and 920-1050°C. Tentative interpretations are presented for the hardness maxima and minima. Annealing of specimens at 1200°C results in significant increase of VHNs upon removal of a 0.05-mm-thickness surface layer from the specimens.
- 6.3.9 **EFFECT OF OXIDATION OF TENSILE BEHAVIOR OF V-5Cr-5Ti ALLOY** – K. Natesan and W. K. Soppet (Argonne National Laboratory). 194
- Oxidation studies were conducted on V-5Cr-5Ti alloy specimens at 500°C in an air environment. The oxidation rates calculated from measurements of thermogravimetric testing are 10, 17, and 25 $\mu\text{m}/\text{y}$ at 400, 450, and 500°C, respectively. Uniaxial tensile specimens were oxidized for several time periods in air at 500°C and subsequently tensile-tested at 500°C in air. The hardened layer in each of these oxidized specimens was confined to 75 μm after 1000 h exposure at 500°C. The influence of 1000 h oxidation is to increase the ultimate tensile strength of the alloy by $\approx 10\%$ while decreasing the tensile rupture strain from 0.23 to 0.14.
- 6.3.10 **EFFECT OF DYNAMICALLY CHARGED HELIUM ON TENSILE PROPERTIES OF V-4Cr-4Ti** – H. M. Chung, B. A. Loomis, L. Nowicki, and D. L. Smith (Argonne National Laboratory). 198
- One property of vanadium-base alloys that is not well understood in terms of their potential use as fusion reactor structural materials is the effect of simultaneous generation of helium and neutron damage under conditions relevant to fusion reactor operation. In the present Dynamic Helium Charging Experiment (DHCE), helium was produced uniformly in the specimen at linear rates of ≈ 0.4 to 4.2 appm helium/dpa by the decay of tritium during irradiation to 18-31 dpa at 425-600°C in the Li-filled DHCE capsules in the Fast Flux Test Facility. This report presents results of postirradiation tests of tensile properties of V-4Cr-4Ti, an alloy identified as the most promising vanadium-base alloy for fusion reactors on the basis of its superior baseline and irradiation properties. Effects of helium on tensile strength and ductility were insignificant after irradiation and testing at $>420^\circ\text{C}$. Contrary to initial expectation, room-temperature ductilities of DHCE specimens were higher than those of non-DHCE specimens (in which there was negligible helium generation), whereas strengths were lower, indicating that different types of hardening centers are produced during DHCE and non-DHCE irradiation. In strong contrast to tritium-trick experiments in which dense coalescence of helium bubbles is produced on grain boundaries in the absence of

displacement damage, no intergranular fracture were observed in any tensile specimens irradiated in the DHCE.

- 6.3.11 DUCTILE-BRITTLE TRANSITION BEHAVIOR OF V-4Cr-4Ti IRRADIATED IN THE DYNAMIC HELIUM CHARGING EXPERIMENT – H. M. Chung, L. J. Nowicki, D. E. Busch, and D. L. Smith (Argonne National Laboratory). 205

One property of vanadium-base alloys that is not well understood in terms of their potential use as fusion reactor structural materials is the effect of simultaneous generation of helium and neutron damage under conditions relevant to fusion reactor operation. In the present DHCE, helium was produced uniformly in the specimen at linear rates ranging from ≈ 0.4 to 4.2 appm helium/dpa by the decay of tritium during irradiation to 18-31 dpa at 425-600°C in Li-filled DHCE capsules in the Fast Flux Test Facility. Ductile-brittle transition behavior of V-4Cr-4Ti, recently identified as the most promising vanadium-base alloy for fusion reactor use, was determined from multiple-bending tests (at -196 to 50°C) and quantitative SEM fractography on TEM disks (0.3-mm thick) and broken tensile specimens (1.0-mm thick). No brittle behavior was observed at temperatures $> -150^\circ\text{C}$, and predominantly brittle-cleavage fracture morphologies were observed only at -196°C in some specimens irradiated to 31 dpa at 425°C during DHCE. Ductile-brittle transition temperatures (DBTTs) were -200°C to -175°C for both types of specimens. In strong contrast to tritium-trick experiments in which dense coalescence of helium bubbles is produced on grain boundaries in the absence of displacement damage, no intergranular fracture was observed in the bend-tested specimens irradiated in the DHCE.

- 6.3.12 VOID STRUCTURE AND DENSITY CHANGE OF VANADIUM-BASE ALLOYS IRRADIATED IN THE DYNAMIC HELIUM CHARGING EXPERIMENT – H. M. Chung, L. Nowicki, J. Gazda, and D. L. Smith (Argonne National Laboratory). . . 211

Combined effects of dynamically charged helium and neutron damage on density change, void distribution, and microstructural evolution of V-4Cr-4Ti alloy have been determined after irradiation to 18-31 dpa at 425-600°C in the DHCE, and the results compared with those from a non-DHCE in which helium generation was negligible. For specimens irradiated to $\approx 18-31$ dpa at 500-600°C with a helium generation rate of 0.4-4.2 appm He/dpa, only a few helium bubbles were observed at the interface of grain matrices and some of the Ti(O,N,C) precipitates, and no microvoids or helium bubbles were observed at the interface of grain matrices and some of the Ti(O,N,C) precipitates, and no microvoids or helium bubbles were observed either in grain matrices or near grain boundaries. Under these conditions, dynamically produced helium atoms seem to be trapped in the grain matrix without significant bubble nucleation or growth, and in accordance with this, density changes from DHCE and non-DHCE (negligible helium generation) were similar for comparable fluence and irradiation temperature. Only for specimens irradiated to ≈ 31 dpa at 425°C, when helium was generated at a rate of 0.4-0.8 appm helium/dpa, were diffuse helium bubbles observed in limited regions of grain matrices and near $\approx 15\%$ of the grain boundaries in densities significantly lower than those in the extensive coalescences of helium bubbles typical of other alloys irradiated in tritium-trick experiments. Density changes of specimens irradiated at 425°C in the DHCE were somewhat higher than those from non-DHCE irradiation. Microstructural evolution in V-4Cr-4Ti was similar for DHCE and non-DHCE except for helium bubble number density and distribution. As in non-DHCE, the irradiation-induced precipitation of ultrafine Ti_5Si_3 was observed for DHCE at $> 500^\circ\text{C}$ but not at 425°C.

6.4 COPPER ALLOYS..... 219

- 6.4.1 EFFECT OF FISSION NEUTRON IRRADIATION ON THE TENSILE AND ELECTRICAL PROPERTIES OF COPPER AND COPPER ALLOYS – S. A. Fabritsiev (D.V. Efremov Institute, St. Petersburg, Russia), A. S. Pokrovsky (SRIAR, Dimitrovgrad, Russia), S. J. Zinkle (Oak Ridge National Laboratory), and D. J. Edwards (Pacific Northwest Laboratory). 221

The tensile and electrical properties of several different copper alloys have been measured following fission neutron irradiation to ~1 and 5 dpa at temperatures between ~90 and 200°C in the SM-2 reactor. These low temperature irradiations caused significant radiation hardening and a dramatic decrease in the work hardening ability of copper and copper alloys. The uniform elongation was higher at 200°C compared to 100°C, but still remained below 1% for most of the copper alloys. As expected, specimens shielded from the thermal neutrons (which produced fusion-relevant solid transmutation rates) exhibited a lower increase in their electrical resistivity compared to unshielded specimens. A somewhat surprising observation was that the radiation hardening was significantly higher in unshielded copper specimens compared to spectrally-shielded specimens.

6.5 ENVIRONMENTAL EFFECTS IN STRUCTURAL MATERIALS..... 229

- 6.5.1 FABRICATION AND PERFORMANCE TESTING OF CaO INSULATOR COATINGS ON V-5%Cr-5%Ti IN LIQUID LITHIUM – J.-H. Park and G. Dragel (Argonne National Laboratory). 231

The electrical resistance of CaO coatings produced on V-5%Cr-5%Ti by exposure of the alloy to liquid Li that contained 0.5-85 wt.% dissolved Ca was measured as a function of time at temperatures between 250 and 600°C. The solute element, Ca in liquid Li, reacted with the alloy substrate at 400-420°C to produce a CaO coating. Resistance of the coating layer measured in-situ in liquid Li was ≈ 0.4 and $6.4 \times 10^6 \Omega$ at 267 and 400°C, respectively. Thermal cycling between 300 and 700°C changed the resistance of the coating layer, which followed insulator behavior. Examination of the specimen after cooling to room temperature revealed cracks in the CaO coating; therefore preliminary tests were conducted to investigate in-situ self-healing behavior. At $\geq 360^\circ\text{C}$, relatively fast healing was indicated. These results suggest that thin homogeneous coatings can be produced on variously shaped surfaces by controlling the exposure time, temperature, and composition of the liquid metal. This coating method is applicable to reactor components. The liquid metal can be used over and over because only the solutes are consumed, not the liquid metal itself. The technique can be applied to various shapes (e.g., inside/outside of tubes, complex geometrical shapes) because the coating is formed by liquid-phase reaction.

- 6.5.2 FABRICATION OF ALUMINUM NITRIDE AND ITS STABILITY IN LIQUID ALKALI METALS – K. Natesan and D. L. Rink (Argonne National Laboratory). 245

AlN has been selected as a prime candidate to electrically insulate the V-alloy first wall in the self-cooled concept for ITER application. Several methods are being evaluated for fabrication of AlN coatings with adequate thickness and the desirable physical, electrical, chemical, and mechanical properties. Coatings developed thus far are being evaluated by exposure to liquid Li at temperatures of 300 to 400°C.

- 6.5.3 CHEMICAL COMPATIBILITY OF STRUCTURAL MATERIALS IN ALKALI METALS – K. Natesan, D. L. Rink, R. Haglund, and R. W. Clark (Argonne National Laboratory). 249

Candidate structural materials are being evaluated with regard to their compatibility, interstitial element transfer, and corrosion in liquid alkali metal systems such as lithium and NaK. Type 316 stainless steel and V-5Cr-5Ti coupon specimens with and without prealuminizing treatment have been exposed to a lithium environment of commercial

purity for 3200 h at 350°C. Weight change data showed negligible corrosion of these materials at this temperature.

7.0 SOLID BREEDING MATERIALS AND BERYLLIUM. 253

- 7.1 AN INVESTIGATION OF THE DESORPTION OF HYDROGEN FROM LITHIUM OXIDE USING TEMPERATURE PROGRAMMED DESORPTION AND DIFFUSE REFLECTANCE INFRARED SPECTROSCOPY – J. P. Kopasz and C. E. Johnson (Argonne National Laboratory) and J. Ortiz-Villafuerte (Escuela Superior de Fisica y Matematicas, Mexico). 255**

A combination of Temperature Programmed Desorption (TPD) and Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) is being used to investigate the desorption of hydrogen from lithium oxide. Initial studies have indicated that there are four different types of hydroxyl groups which can be observed on a lithium oxide surface. The particular species present vary depending on the temperature and hydrogen pressure of the system. Under some conditions where hydrogen is present in the purge gas surface hydride species have been observed. This suggests heterolytic adsorption of hydrogen has occurred.

8.0 CERAMICS. 265

- 8.1 FATIGUE CRACK GROWTH RATE (FCGR) BEHAVIOR OF NICALON/SiC COMPOSITES – N. Miriyala, P. K. Liaw, N. Yu, and C. J. McHargue (University of Tennessee), L. L. Snead (Oak Ridge National Laboratory), and D. K. Hsu (Iowa State University). 267**

Ultrasonic measurements were continued on the Nicalon/SiC composite specimens to correlate elastic moduli with percentage porosity in the in-plane as well as through-thickness directions. A micromechanics model based on periodic microstructure was developed to predict the elastic stiffness constants of the Nicalon/SiC composites. The predicted values were in good agreement with the experimental results.

- 8.2 ADVANCED SiC COMPOSITES FOR FUSION APPLICATIONS – L. L. Snead and O. J. Schwarz (Oak Ridge National Laboratory). 274**

Chemically vapor infiltrated silicon carbide (SiC) composites have been fabricated from continuous fibers of either SiC or graphite and tested for strength and thermal conductivity. Of significance is that the Hi-NicalonTM SiC based fiber composite has superior unirradiated properties as compared to the standard Nicalon grade. Based on previous results on the stability of the Hi-Nicalon fiber, this system should prove more resistant to neutron irradiation. A graphite fiber composite has been fabricated with very good mechanical properties and thermal conductivity an order of magnitude higher than typical SiC/SiC composites.

- 8.3 THERMAL CONDUCTIVITY DEGRADATION OF GRAPHITES IRRADIATED AT LOW TEMPERATURE – L. L. Snead and T. D. Burchell (Oak Ridge National Laboratory). 289**

Several graphites and graphite composites (C/C's) have been irradiated near 150°C and at fluences up to a displacement level of 0.24 dpa. The materials ranged in unirradiated room temperature thermal conductivity varied from 114 W/m-K for H-451 isotropic graphite, to 670 W/m-K for unidirectional FMI-1D C/C composite. At the irradiation temperature a saturation reduction in thermal conductivity was seen to occur at displacement levels of approximately 0.1 dpa. All materials were seen to degrade to approximately 10 to 14% of their original thermal conductivity after irradiation. The effect of postirradiation annealing on the thermal conductivity was also studied.

- 8.4 INCUBATION TIME FOR SUB-CRITICAL CRACK PROPAGATION IN SiC-SiC COMPOSITES – A. El-Azab and N. M. Ghoniem (University of California, Los Angeles). 296

The effects of fiber thermal creep on the relaxation of crack bridging tractions in SiC-SiC ceramic matrix composites (CMCs) is considered in the present work, with the objective of studying the time-to propagation of sub-critical matrix cracks in this material at high temperatures. Under the condition of fiber stress relaxation in the bridging zone, it is found that the crack opening and the stress intensity factor increase with time for sub-critical matrix cracks. The time elapsed before the stress intensity reaches the critical value for crack propagation is calculated as a function of the initial crack length, applied stress and temperature. Stability domains for matrix cracks are defined, which provide guidelines for conducting high-temperature crack propagation experiments.

- 8.5 APPARENT ACTIVATION ENERGY OF SUBCRITICAL CRACK GROWTH OF SiC/SiC COMPOSITES AT ELEVATED TEMPERATURES – Y. S. Chou (Associated Western Universities, NW), N. M. Stackpoole and R. Bordia (University of Washington, Seattle), C. H. Henager, Jr., C. F. Windisch, Jr., and R. H. Jones (Pacific Northwest Laboratory). 311

In the past six months, we have conducted studies of subcritical crack growth on SiC/SiC composite materials in a corrosive (O₂) as well as an inert (Ar) atmosphere for temperatures ranging from 800 to 1100°C. Two materials, one with ~1 μm carbon (C) interface and the other with ~0.5 μm boron nitride (BN), were investigated. Apparent activation energies (E_{act}) were determined from both the crack velocity and thermogravimetric analysis. In pure Ar, it was found that the apparent activation energy gradually increased with time, consistent with the development of steady-state bridging zone. The asymptotic value for E_{act} from crack growth data was found to be ~205 kJ/mol and ~234 kJ/mol for BN- and C-interface materials, respectively, in good agreement with published data (~200 kJ/mol) for creep of Nicalon fibers. In the presence of oxygen, E_{act} decreased to ~40-50 kJ/mol for C-interface and ~50-68 kJ/mol for BN-interface. Microstructural characterization of the oxidized samples indicated that the growth rate of the reaction front for BN-interface materials is an order of magnitude lower than for C-interface ones. At higher temperatures, a glassy phase was observed to seal off the BN-interface, whereas the C-interface remained open during all tests.

- 8.6 EFFECTS OF NEUTRON IRRADIATION ON DIMENSIONAL STABILITY AND ON MECHANICAL PROPERTIES OF SiC/SiC COMPOSITES – G. E. Youngblood, G. H. Henager, Jr., D. J. Senor, and G. W. Hollenberg (Pacific Northwest Laboratory). . 321

The dimensional stability and some mechanical properties of two similar 2D 0-90° weave SiC_f/SiC composite made with NicalonTM ceramic-grade (CG) fiber were characterized and compared after neutron irradiation to those properties for β-SiC. The major difference between these two composites was that one had a thin (150 nm) and the other a thick (1000 nm) graphite interface layer. The irradiation conditions consisted of relatively high doses (4.3 to 26 dpa-SiC) at high temperatures (430 to 1200°C).

Up to about 900°C, swelling of the irradiated SiC_f/SiC composites (<0.5%) was slightly less than for irradiated monolithic SiC and was relatively independent of dose. The strengths and the modulus of these SiC_f/SiC composites were reduced by about 50% by the irradiation. During irradiation, the Nicalon CG fibers tended to densify and shrink, thus partially decoupling the fibers from the matrix. The decoupling of the fibers from the matrix led to loss of load transfer capability and effectively increased the porosity of the material. Considerable microcracking of the matrix also resulted due to the residual stresses between the shrinking fibers and the expanding matrix.

Synthesis of irradiation resistant SiC_f/SiC composites in the future will require fabrication using improved SiC fibers with better irradiation damage stability. Only then

can the fiber/matrix interface thickness and perhaps type be optimized for better performance.

- 8.7 EFFECT OF IRRADIATION SPECTRUM ON THE MICROSTRUCTURAL EVOLUTION IN CERAMIC INSULATORS – S. J. Zinkle (Oak Ridge National Laboratory) 331

Cross section transmission electron microscopy has been used to investigate the microstructure of $MgAl_2O_4$ (spinel) and Al_2O_3 (alumina) following irradiation with ions of varying mass and energy at room temperature and 650°C. Dislocation loop formation was suppressed in specimens irradiated with light ions, particularly in the case of spinel. An evaluation of the data showed that dislocation loop formation during irradiation at 650°C was suppressed when the ratio of the electronic- to nuclear-stopping power was greater than ~10 and ~1000 for spinel and alumina, respectively. The effect of uniform background levels of ionizing radiation on the microstructural evolution in spinel was investigated by performing simultaneous dual-beam (He^+ and heavy ion) irradiations. The uniform ionizing radiation source did not affect the microstructural evolution of spinel unless the ionization was very intense (average electronic- to nuclear-stopping power ratio >100). These results clearly indicate that light ion and electron irradiations produce microstructures which are not representative of the microstructure that would form in these ceramics during fission or fusion neutron irradiation.

- 8.8 DATA ACQUISITION SYSTEM USED IN RADIATION INDUCED ELECTRICAL DEGRADATION EXPERIMENTS – D. P. White (Oak Ridge National Laboratory) 346

It has been observed that some oxide ceramics which have an electric field applied to them while simultaneously being irradiated may undergo a degradation of their insulating properties under certain conditions. An *in-situ* DC conductivity capsule has been constructed to study the effect of neutron irradiation on the electrical conductivity of alumina with an applied electric field at the HFBR. The current capsule differs from a previous design in that the current measurements are performed on the low voltage side of the circuit.

The data acquisition system to be described here has been built and will be used to perform the electrical conductivity measurements in an experiment scheduled to begin in October 1994.

- 8.9 CATION DISORDER IN HIGH-DOSE, NEUTRON-IRRADIATED SPINEL – K. E. Sickafus, A. C. Larson, N. Yu, M. Nastasi (Los Alamos National Laboratory), G. W. Hollenberg and F. A. Garner (Pacific Northwest Laboratory), and R. C. Bradt (University of Nevada-Reno) 350

The crystal structures of $MgAl_2O_4$ spinel single crystals irradiated to high neutron fluences [$5 \cdot 10^{26}$ n/m² ($E_n > 0.1$ MeV)] were examined by neutron diffraction. Crystal structure refinement of the highest dose sample indicated that the average scattering strength of the tetrahedral crystal sites decreased by ~20% while increasing by ~8% on octahedral sites. Since the neutron scattering length for Mg is considerably larger than for Al, this result is consistent with site exchange between Mg^{2+} ions on tetrahedral sites and Al^{3+} ions on octahedral sites. Least-squares refinements also indicated that, in all irradiated samples, at least 35% of Mg^{2+} and Al^{3+} ions in the crystal experienced disordering replacements. This retained dpa on the cation sublattices is the largest retained damage ever measured in an irradiated spinel material.