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1.1 STUDY OF THE LONG-TERM STABILITY OF MHD COATINGS FOR FUSION REACTOR APPLICATIONS – B. A. Pint, L. D. Chitwood, J. H. DaVan, and J. R. DiStefano (Oak Ridge National Laboratory 3

Bulk specimens of two candidate compositions, CaO and AlN, for insulating coatings in a lithium-cooled fusion reactor have been exposed to lithium in 1000h isothermal tests at 500°-800°C to determine the maximum temperature at which acceptable compatibility is likely. Specimens of AlN+0.04Y showed significant mass loss at 700° and 800°C but much less attack at 600°C. For\ testing at 600°C, changing from a V alloy capsule liner to a Mo liner or adding 1000 ppm N to the Li caused only minor differences in the mass change. Polished cross-sections indicated that a reaction layer had formed in each case. Single crystals of CaO showed massive losses at 600° and 700°C similar to results with polycrystalline specimens. With the addition of 1000 ppm O to the Li, there was a change in the amount of CaO dissolved during the 1000h exposure suggesting that doping Li may be an avenue for improved compatibility. All of these results are consistent with an analysis of the relevant thermodynamic solubility data.

1.2 DEVELOPMENT AND CHARACTERIZATION OF ELECTRICALLY INSULATING CaO COATINGS — K. Natesan, M. Uz, D. L. Rink, and D. L. Smith (Argonne National Laboratory) 9

As part of the Department of Energy's Fusion Reactor Program, studies have been in progress at Argonne National Laboratory (ANL) to develop electrically insulating coatings on V-Cr-Ti alloys, in particular, on V- 4Cr-4Ti which is the primary candidate for use in various structural applications, including in the first wall structure/blanket of a fusion reactor. The first wall will be in contact with liquid Li coolant and electrical conductivity across the wall will lead to magnetohydrodynamic, pressure losses during flow in the magnetic field. Hence, among the critical requirements of the rather stringent design criteria for the first wall material are that the coating on it be non-porous, tenacious, electrically insulating, and capable of maintaining its structural integrity while in use in a liquid Li environment at temperatures of 400-700°C. More information on the material requirements of fusion reactors is reported in an earlier publication. This report addresses the development and characterization of a CaO coating that was applied on V-4Cr-4Ti alloy by a vapor transport (or thermal/chemical vapor deposition) process. Several coupon and rod specimens were coated with CaO by a double Ca-deposition/oxidation process that was developed at ANL. The specimens were analyzed and the coating was characterized after each step of the process as well as before and after exposure to a liquid Li environment. The analysis and characterization involved the use of one or more of scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX), and X-ray diffraction (XRD) techniques. Results are presented, with emphasis on microstructural analysis of the samples and on the details of the developed experimental procedure.

1.3 STRUCTURAL ANALYSIS OF PLATE-SHAPED PRECIPITATES IN NEUTRON IRRADIATED V-4Cr-4Ti — D. T.Hoelzer and S. J Zinkle (Oak Ridge National Laboratory) 19

Neutron irradiation of V-4Cr-4Ti t ~510 °C and dose of ~4 dpa resulted in the formation of relatively high number density of plate shaped precipitates on {100}bcc habit planes.The TEM analysis showed that their average size was ~24 nm (+/-12 nm)in diameter and ~1-3 nm in thickness.Diffuse streaking from the precipitates was observed to lie parallel to <100>bcc directions and centered at 3 /4 <200>positions in electron diffraction patterns.Based on an orientation relationship observed between the precipitates and bcc matrix and a tilting experiment on one plate variant,the structural analysis indicated that the crystal structure of the plates was consistent with the fcc structure,which is the same for the golobular-shaped Ti-(OCN)phase. The analysis also showed that diffuse scattering observed at 2/3 <222>positions in electron diffraction patterns is not caused by the plate shaped phase.

1.4 HELIUM ANALYSIS FROM THE DHCE-1 SIMULATION EXPERIMENT — D. L. Smith (Argonne National Laboratory) 26

A detailed calculation of the predicted helium generation rates in unalloyed vanadium and the reference V-4Cr-4Ti alloy irradiated in the DHCE-1 Proof-of-Principle experiment has been performed with the available data base on fundamental properties. Results of these detailed calculations indicate that the experimentally measured helium concentrations are in good agreement with the calculated values. Approximately 90% of the measured values are within a factor of 0.5 to 1.5 of the calculated values, which is quite good agreement. The validity of the experiment is further verified by a comparison of the experimental results from the other alloys included in the experiment with calculated correlation factors for each of the seven capsules. This paper presents a summary of the comparison of the results from the detailed calculations with experimental helium concentrations in the vanadium alloys in the DHCE-1 proof-of-principle experiment.

1.5 QUANTITATIVE OXYGEN ANALYSES FOR V-4Cr-4Ti ALLOYS — Y. Yan, D. P. McGann, D. L. Smith, and H. Tsai (Argonne National Laboratory) 34

A quantitative oxygen analysis has been performed for V-4Cr-4Ti alloys. The oxygen concentration is about 210 wppm for Japan's NIFS-1 heat, 324 wppm for ANL's 832665 heat, and 385 wppm for the GA's 832864 heat. Our experiment indicates that, after cutting, the oxygen analysis specimen should be pickled to remove the surface oxide.

1.6 UNIAXIAL CREEP BEHAVIOR OF V-4Cr-4Ti ALLOY — K. Natesan, W. K. Soppet, and D. L. Rink (Argonne National Laboratory) 37

A systematic study is currently being conducted at Argonne National Laboratory (ANL) to evaluate the uniaxial creep behavior of V-Cr-Ti alloys as a function of temperature in the range of 650-800°C and at applied stress levels in the range of 75-380 MPa. At present, the principal effort has focused on the V-4Cr-4Ti alloy of Heat 832665; however, another heat of a similar alloy from General Atomics (GA) will also be used in the study. During this reporting period, additional creep tests were

conducted at 700 and 800°C at lower stress levels than in earlier tests. In addition, the tested specimens are examined for O contamination, if any, by vacuum fusion analysis of the tested specimens and by micro hardness measurements of specimen cross-sections. The test results indicate that in the temperature range of 650-800°C, creep deformation follows a power-law creep with stress exponents indicative of a dislocation-climb-controlled process.

1.7 CHARACTERIZATION AND IMPACT PROPERTIES OF V-4Cr-4Ti LASER WELDMENTS* — Y. Yan, D. L. Smith, Z. Xu, and H. Tsai, (Argonne National Laboratory) and T. Nagasaka and T. Muroga (National Institute of Fusion Science, Japan) 41

The effects of alloy chemistry and microstructure on the Charpy-impact properties of laser weldments of three heats of V-4Cr-4Ti alloy were investigated. The impact properties of all three heats indicate similar behavior with a DBTT of approximately –200°C based on one-third size Charpy specimens in the annealed condition. However, The Charpy impact properties of the weldments, which have a significantly differentmicrostructure, indicate significantly different DBTT's for the three heats. Laser welds or bead-on-plate microstructures obtained with the laser beam provide a unique method for investigating the effects of microsturctural and trace element concentrations on the properties of these alloys. Chemical analyses for several trace elements have been performed on the three heats in an attempt to determine which elements affect the mechanical properties. Further research is in progress to investigate the sensitivity of the V-4Cr-4Ti alloy system to minor variations in trace element concentrations and microstructural variations produced by varying the heat treatment.

1.8 MICROSTRUCTURAL EXAMINATION OF IRRADIATED V-4CR-4TI PRESSURIZED CREEP TUBES — D. S. Gelles (Pacific Northwest National Laboratory) 45

Three pressurized tubes of V-4Cr-4Ti have been examined to determine microstructural development due to irradiation creep following irradiation in ATR at 300°C.

1.9 IMPACT PROPERTIES OF V-4Cr-4Ti LASER WELDMENTS FROM THE GA'S HEAT — Y. Yan, H. Tsai, A. D. Storey, D.L. Smith, and Z. Xu (Argonne National Laboratory) 50

Charpy-impact properties of laser weldments of GA heat 832864 V-4Cr-4Ti alloy were investigated. Impact testing was performed with annealed specimens of the both base metal and the laser weldment in as-machined (by electric discharge machining with water as the flushing fluid) condition. The ductile-to-brittle-transition temperature (DBTT) for the base metal is below –187°C and for the laser weld is about 0°C. Additional microstructure characterization on the laser weld is underway.

1.10 SUMMARY REPORT ON THE US/JAPAN JUPITER COLLABORATION INTEGRATED FOCUS ON FUNDAMENTAL STUDIES – VANADIUM INITIATIVE – H.L. Heinisch (Pacific Northwest National Laboratory •) and N. Sekimura (University of Tokyo) 55

IFFS-VI was initiated by the participants in the JUPITER Workshop on Theory and Modeling for Fusion Materials held October 30, 1997, during ICFRM-8 in Sendai, Japan. The original initiative is described, participants and collaborations are identified, and progress is summarized.

2.0 SILICON CARBIDE COOMPOSITE MATERIALS 62

2.1 EFFECT OF HEAT TREATMENT ON SILICON CARBIDE BASED JOINING MATERIALS FOR FUSION ENERGY — C. A. Lewinsohn and R. H. Jones (Pacific Northwest National Laboratory)• , T. Nozawa, M. Kotani, H. Kishimoto, Y. Katoh and A. Kohyama (Kyoto University, Japan). 63

Two general approaches to obtaining silicon carbide-based joint materials were used. The first method relies on reactions between silicon and carbon to form silicon carbide, or to bond silicon carbide powders together. The second method consists of pyrolysing a polycarbosilane polymer to yield an amorphous, covalently bonded material. In order to assess the long-term durability of the joint materials, various heat treatments were performed and the effects on the mechanical properties of the joints were measured. Although the joints derived from the polycarbosilane polymer were not the strongest, the value of strength measured was not affected by heat treatment. On the other hand, the value of the strength of the reaction-based joints was affected by heat treatment, indicating the presence of residual stresses or unreacted material subsequent to processing. Further investigation of reaction-based joining should consist of detailed microscopic studies; however, continued study of joints derived from polymers is also warranted.

2.2 MECHANICAL PROPERTIES OF HELIUM IMPLANTED SILICON CARBIDE- L L Snead (Oak Ridge National Laboratory), R. Scholz (JRC Ispra), A. Frias Rebelo (Whereabouts Unknown) 69

It has been long apparent that ceramics will undergo significant gas production, transmutation and material "burn-up" under high energy neutron irradiation. For example, the original calculations for pure SiC exposed to 1 MW-a/m^2 of 14.1 MeV monoenergetic neutrons yielded 1596 appm helium, 440 appm hydrogen, 458 appm magnesium, 234 appm beryllium and 72 appm aluminum, as well as some less significant transmutants. Clearly, helium is the largest product and, because of its limited diffusivity in SiC, may cause significant swelling and/or stress in the material. This paper will give preliminary results of the effect of high levels of helium on the mechanical properties of CVD SiC.

2.3 TENSILE TESTING OF UNIDIRECTIONAL SILICON CARBIDE COMPOSITES FOR FUTURE IRRADIATION EXPERMENTS— 74
T. Hinoki,[1] L.L. Snead,[2] E. Lara-Curzio,[2] Y. Katoh,1 and A. Kohyama[1] 1
(1) Institute of Advanced Energy, Kyoto University; (2) Oak Ridge National Laboratory

The SiC/SiC composites, which have three kinds of unidirectional stoichiometric SiC fibers and various fiber coating, were prepared by I-CVI method for future irradiation experiments. In-plane tensile bars and transthickness tensile specimens were cut from these materials. In-plane tensile tests, transthickness tensile tests and four point bend tests were carried out at ambient temperature. While the specimens, SCS-9A™ fibers were used, showed superior ultimate tensile stress, more than 1 GPa, to the other specimens, proportional limit stress of the specimens, Hi-Nicalon™ type-S fibers were used, was larger than the other specimens. The specimens, Tyranno™ SA fibers were used or multiple SiC fiber coating was applied, showed brittle fracture behavior. Correlation between tensile results and flexural results was discussed. Transthickness tensile tests were unsuccessful because of insufficient bond between fixture and epoxy.

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3.1 SUMMARY OF IEA WORKSHOP/WORKING GROUP MEETING ON FERRITIC/MARTENSITIC STEELS FOR FUSION — R. L. Klueh 86
(Oak Ridge National Laboratory)

The International Energy Agency (IEA) Working Group on Ferritic/Martensitic Steels for Fusion held a workshop in Tokyo, Japan, 2-3 November 2000. Participants came from Europe, Russia, the United States, and Japan to review the progress of the collaboration on reduced-activation ferritic/martensitic steels and the development of plans for future collaboration. At this workshop, data that have been obtained during the collaboration were reviewed, and it was generally agreed that the testing phase for the IEA heats of steel obtained for this collaboration is near completion. A database has been established on the completed work, and that database is available to the international community. Based partially on the work performed in this collaboration, the European Union has developed specifications for a new reduced-activation steel, EUROFER 97, which has been produced and is now being evaluated. The other material of interest at this workshop was oxide dispersion-strengthened (ODS) steel, which was the subject of presentations from Europe, Japan, and the United States. The consensus is that selective utilization of these steels offers the possibility of a ferritic martensitic steel that will allow higher operating temperatures for a fusion device, although it is recognized that ODS steels are still in the early stage of development.

3.2 RECENT RESULTS FOR THE FERRITICS ISOTOPIC TAILORING (FIST) EXPERIMENT — D. S. Gelles, M. L. Hamilton, B. M. Oliver, and L. R. Greenwood 93
(Pacific Northwest National Laboratory), S. Ohnuki (University of Hokkaido, Japan), K. Shiba (JAERI Tokai, Japan), Y. Kohno (Muroran Institute of Technology, Japan), A. Kohyama (Kyoto University, Japan) and J. P. Robertson (Oak Ridge National Laboratory)

The results of shear punch testing, microstructural examination, and hydrogen and helium analyses performed on irradiated isotopically tailored alloys are reported for specimens irradiated in the HFIR JP20 experiment.

3.3 SHEAR PUNCH PROPERTIES OF LOW ACTIVATION FERRITIC STEELS FOLLOWING IRRADIATION IN ORR — R. M. Ermi, M. L. Hamilton and D. S. Gelles (Pacific Northwest National Laboratory) and A. M. Ermi (COGEMA Engineering Corporation, Richland, WA) 103

Shear punch post-irradiation test results are reported for a series of low activation steels containing Mn following irradiation in the Oak Ridge Reactor at 330 and 400°C to ~10 dpa. Alloy compositions included 2Cr, 9Cr and 12Cr steels with V to 1.5% and W to 1.0%. Comparison of results with tensile test results showed good correlations with previously observed trends except where disks were improperly manufactured because they were too thin or because engraving was faulty.

3.4 LONG-TERM HIGH TEMPERATURE OXIDATION BEHAVIOR OF ODS FERRITICS — B. A. Pint (Oak Ridge National Laboratory) 107

Four ODS ferritic compositions were tested for up to 10,000h at 700°-1100°C. At 700°-800°C in air, the reaction rates were very low for all of the alloys. At 900°C, the addition of Y_2O_3, compared to Al_2O_3, showed a distinct benefit in improving the oxidation resistance, due to a reactive element effect. The absence of Ti and W in one alloy appeared to result in a thinner reaction product after oxidation at 800°C. One composition was tested in 10% water vapor at 900°C and at 1000°C and 1100°C in air. Under these higher temperature conditions, there was a significant increase in the rates of oxidation. With only 13-14at%Cr in these alloys, their corrosion-limited operating temperature is less than 1000°C.

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No contributions.

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5.1 IMPROVEMENTS IN THE DUCTILITY OF MOLYBDENUM WELDMENTS BY ALLOYING ADDITIONS OF Zr, B and C — M. K. Miller*, E. A. Kenik*, K. F. Russell (Oak Ridge National Laboratory) and A. J. Bryhan (Applied Materials, Santa Clara, CA) 116

A significant improvement in the ductility of molybdenum weldments has been achieved through the addition of zirconium, aluminum, carbon, and boron at the parts per million level. A ductility of 20% has been obtained in gas-tungsten arc weldments in 6.35-mm-thick plate. This improvement over molybdenum's traditional 3% ductility has been achieved by improving the normally low fracture stress of grain boundaries. Atom probe tomography has revealed segregation of zirconium, boron and carbon to and the depletion of oxygen at the grain boundaries in the base metal and the heat affected zone.

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6.1 VOID SWELLING AT LOW DISPLACEMENT RATES IN ANNEALED X18H10T STAINLESS STEEL AT 4 to 56 dpa AND 280-332°C 123
F. A. Garner (Pacific Northwest National Laboratory*), S. I. Porollo, A. N. Vorobjev, Yu. V. Konobeev, and A. M. Dvoriashin (Institute of Physics and Power Engineering, Russia)

Various components of pressurized water power reactors (PWRs) and some proposed fusion devices such as ITER will operate at lower temperatures and displacement rates than are encountered in many test reactors such as EBR-II, FFTF and HFIR. The question arises if the presence and magnitude of void swelling can be predicted for such irradiation environments. Data on Russian steel can be used to address part of this question. In reactor applications where Western countries typically use annealed AISI 304 stainless steel, it is the Russian practice to use annealed X18H10T, a titanium-stabilized 18Cr-10Ni stainless steel analogous to AISI 321. Using a flow restrictor component from the low-flux breeder zone of the BN-350 reactor in Kazakhstan, it was possible to examine the behavior of void swelling at relatively low temperatures and low displacement rates after 12 years of irradiation. The temperature of this component ranged from 270-340°C with a peak dose rate of 1.6×10^{-7} dpa/sec and a peak dose of 56 dpa. Careful sectioning of the component has yielded a large number of microscopy specimens over a ITER-relevant range of temperatures and displacement rates. Microstructural data are presented and show that void swelling at 10 to 50 dpa persists down to ~306°C for dose rates on the order of 1×10^{-7} dpa/sec.

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7.1 MICROSTRUCTURE OF SWIFT HEAVY ION IRRADIATED SiC, Si_3N_4 AND AlN – S. J. Zinkle, J.W. Jones (Oak Ridge National Laboratory) and V.A. Skuratov (Flerov Laboratory, Dubna, RF) 136

Cross-section transmission electron microscopy was used to investigate the microstructure of single crystal silicon carbide and polycrystalline silicon nitride and aluminum nitride following room temperature irradiation with either 245 MeV Kr or 710 MeV Bi ions. The fluences ranged from $1x10^{12}/cm^2$ (single track regime) to $1x10^{13}/cm^2$. Ion track formation was observed in the Bi ion-irradiated Si_3N_4 specimen in regions where the electronic stopping power exceeded a critical value of ~15 keV/nm (depths <24 µm). Ion track formation was not observed at any depth in 245 MeV Kr ion-irradiated Si_3N_4, in which the maximum electronic stopping power was 14.5 keV/nm. There was no evidence for track formation in either SiC or AlN irradiated with 710 MeV Bi ions, which indicates that the threshold electronic stopping power for track formation in these two ceramics is >34 keV/nm. The high resistance of SiC and AlN to track formation may be due to their high thermal conductivity, but further study is needed to quantitatively evaluate the suitability of the various track formation models.

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9.2 THE EFFECT OF TEST MACHINE COMPLIANCE ON THE MEASURED SHEAR PUNCH YIELD STRESS AS PREDICTED USING FINITE ELEMENT ANALYSIS — Mychailo B. Toloczko (Pacific Northwest National Laboratory, Katsunori Abe (Tohoku University, Japan), Margaret L.Hamilton, Frank A. Garner, and Richard J.Kurtz (Pacific Northwest National Laboratory) 148

In previous research involving the use of the shear punch test,it was assumed that the displacement of the punch tip was only slightly different than the crosshead displacement. The present work explores this assumption and its ramifications by simulating the shear punch test with finite element analysis (FEA).The simulations suggest that punch tip displacement is much less than previously assumed,and that for the test frames which have been used,crosshead displacement is over an order of magnitude greater than punch tip displacement.This difference in displacements is thought to be due to test machine and punch compliance,and a simple elasticity calculation of the compliance of the punch,the test machine,and a specimen gives a result which is in agreement with the FEA simulations. The effect of using punch tip displacement on the observed effective shear yield stress was evaluated using FEA simulated shear punch tests on several different metals.Yield was measured at several different offset shear strains with a 1.0%offset shear yield strength measurement providing the best correlation with 0.2%offset uniaxial yield strength.When using the 1.0%offset shear yield values,the previously observed material-to-material variability in the tensile-shear correlation all but disappeared.Based on this work,it appears that the material-to-material variations in prior correlations between uniaxial yield strength and shear yield strength is due to a combination of large test machine compliance and material-to-material differences in the work hardening exponent.

9.3 CORRELATION OF NANOINDENTATION AND CONVENTIONAL MECHANICAL PROPERTY MEASUREMENTS — P. M. Rice (IBM Almaden Research Center) and R. E. Stoller (Oak Ridge National Laboratory) 160

Extended Abstract.

9.4 EFFECT OF PERIODIC TEMPERATURE VARIATIONS ON THE MICROSTRUCTURE OF NEUTRON-IRRADIATED METALS- S.J. Zinkle, N. Hashimoto, D.T. Hoelzer, A.L. Qualls (Oak Ridge National Laboratory) and T. Muroga (National Institute for Fusion Science) 162

Specimens of pure copper, a high-purity austenitic stainless steel, and V-4Cr-4Ti were exposed to eight cycles of either constant temperature or periodic temperature variations during neutron irradiation in the High Flux Isotopes Reactor to a cumulative damage level of 4 to 5 displacements per atom. Specimens were exposed to a low temperature during the initial 10% of accrued dose in each of the eight cycles, and were exposed to a higher temperature during the remaining 90% of accrued dose in each cycle. Different specimens were exposed to low/high irradiation temperatures of 225/340˚C and 350/520˚C. The microstructure was compared with that of companion

specimens that were continuously maintained at 340˚C and 520˚C, respectively during the entire irradiation. The low-temperature excursions produced enhanced nucleation and growth of radiation-induced defects (precipitates, dislocation loops) in V-4Cr-4Ti and stainless steel.

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No contributions.

11.0 MATERIALS ENGINEERING AND DESIGN REQUIREMENTS 169

No contributions.

12.0 IRRADIATION FACILITIES AND TEST MATRICES 170

No contributions.