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The estimated mid-core total peak fast fluence was 6.85×10^{22} n/cm² ($E > 0.111$ MeV), and the estimated total peak displacements per atom was 32.6 dpa (in stainless steel). During reactor operation, the inlet coolant remained at 371°C while the calculated coolant outlet temperature was 441°C.

1.6 NEUTRONICS ASPECTS OF THE DESIGN OF THE A1 DROP-IN EXPERIMENT
AT ATR – I. C. Gomes and D. L. Smith (Argonne National Laboratory). 79

The transmutation rate of vanadium to chromium was controlled with the use of thermal neutron absorber (gadolinium) which was incorporated, by design, within the drop-in experimental capsule. Gadolinium was selected as the filtering material due to several reasons, among those one can mention, high cross section for thermal neutrons, required thickness to survive 5 dpa's irradiation of less than 2 mm, good neutronics data base, easy handling, and overall cost.

The nuclear heat deposition, accounting for gamma-ray and neutron heating from the surroundings (core and reflector region) and gamma-ray heating produced by the capture of thermal neutrons at the thermal neutron filter, was estimated at each region of the capsule. The temperature distribution inside the capsule was analyzed as a function of the gas gap between the sub-capsule and the holder (maintained at the coolant temperature 60°). The results presented here are considered preliminary, and a more precise estimation of the values is underway.

1.7 VANADIUM ALLOY IRRADIATION EXPERIMENT ATR-A1 IN THE ADVANCED
TEST REACTOR – H. Tsai, R. V. Strain, I. Gomes, A. G. Hins, and D. L. Smith
(Argonne National Laboratory). 81

A collaborative DOE/Monbuscho irradiation experiment is being implemented to generate low-temperature mechanical properties data on vanadium alloys and low-activation ferritic steels. Monbuscho is supplying the latter specimens. The experiment will be conducted in the Advanced Test Reactor at the Idaho National Engineering Laboratory and is designated ATR-A1. The core position selected, Channel A10, has relatively high fast neutron flux and fast-to-thermal flux ratio. These qualities are important for achieving a reasonable damage rate in the specimens and reducing the thickness requirements of thermal neutron filters. Filtering out the thermal neutron flux is necessary in the water-cooled ATR in order to avoid excessive V(n, γ)Cr transmutation.

The test vehicle will consist of four capsule segments containing a total of 15 subcapsules: 13 for vanadium alloy specimens and two for low-activation ferritic steel specimens. In all subcapsules the specimens will be lithium bonded to provide uniform specimen temperature, maximum heat transfer, and, in the case of vanadium alloy specimens, impurity control. Two test temperatures are planned: 200 and 300°C. They will be achieved by filling the gas-gap between the subcapsules and capsule with different blends of He and Ar. The vanadium alloy test specimens will be biaxial creep (pressurized tubes), Charpy impact, compact tension, tensile, and transmission electron microscope (TEM) disks. For the ferritic steels, the specimens will be Charpy impact, tensile, and TEM disks. The goal fluences for the experiment is 5 dpa (in vanadium), which will be attained in ≈ 135 effective full power days (EFPDs) in the A10 position.

Most of the test vehicle design has been completed and fabrication of some of the components is underway. The irradiation is scheduled to begin in August 1995 and be completed in January 1996.

- 1.8 STATUS OF VANADIUM ALLOY IRRADIATION EXPERIMENT X530 IN EBR-II – H. Tsai, R. V. Strain, A. G. Hins, H. M. Chung, L. J. Nowicki, and D. L. Smith (Argonne National Laboratory). 85
- To obtain early irradiation performance data on the new 500-kg production heat of the V-4Cr-4Ti material before the scheduled EBR-II shutdown, an experiment, X530, was expeditiously designed and assembled. Charpy, compact tension, tensile and TEM specimens with different thermal mechanical treatments (TMT) were enclosed in twelve subcapsules and irradiated in the last run of EBR-II, Run 170A. The accrued exposure was 35 effective full power days, yielding a peak damage of ≈ 4 dpa in the specimens. In this reporting period, the irradiation vehicle was disassembled at the Hot Fuel Examination Facility (HFEF) at ANL-West and the subcapsules shipped to ANL-East for disassembly. Subcapsule disassembly is scheduled to be completed at ANL-East in the next reporting period.
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- 2.1 ACTIVATION OF SILICON CARBIDE IN FUSION REACTORS – H. L. Heinisch (Pacific Northwest Laboratory). 91
- Because of production of ^{26}Al from Si, SiC irradiated in a fusion energy system first wall exceeds the limits for shallow land burial, based on 10 CR 61, Class C, for irradiation doses typical of a first wall component service lifetime in DEMO, 12.5 MW y/m². However, if first wall activities can be averaged over entire components that include portions within the fusion machine where fluxes of high energy neutrons are smaller than at the first wall, production of ^{26}Al may stay under the shallow land burial limit for practical component service lifetimes. Realistic information on energy system design, waste disposal criteria, and decommissioning procedures is necessary to determine with certainty the role SiC can play as a low activation fusion energy system material. Sequential charged particle reactions have no significant effect on the residual radioactivity of SiC irradiated in a fusion energy system first wall.
- 2.2 CLEAN STEELS FOR FUSION – D. S. Gelles (Pacific Northwest Laboratory). 97
- A summary of the workshop Clean Steels - Super Clean Steels is provided and a paper given at the Workshop entitled Clean Steels for Fusion is reproduced. The workshop demonstrated, based on ten years of steel making practice, that control of minor impurities, P, Sb, Sn, and As, along with Mn and Si, could effectively eliminate temper embrittlement in 3.5NiCrMoV rotor steels.
- 2.3 THE EFFECTS OF IMPURITIES ON THE ACTIVATION OF SiC, VANADIUM AND FERRITIC ALLOYS – H. Attaya and D. Smith (Argonne National Laboratory). 107
- Consistent transport and activation calculations have been performed to compare the activation responses of the leading reduced-activation materials in a fusion power reactor. Another set of calculations has been made to evaluate the effects of the trace elements on these responses. The materials considered in this work are the V4Cr4Ti vanadium alloy, the 9Cr2WVTa ferritic alloy, and the silicon carbide (SiC). In addition, calculations have also been made for the conventional 316SS and HT-9 alloys. The TPSS conceptual design has been utilized in this work. The results show that the V4Cr4Ti alloy has the minimum operational and the minimum long-term radioactivity and decay heat. The SiC has the minimum intermediate-term radioactivity. The effects of impurities are noticed with respect to the V4Cr4Ti and SiC. With respect to the 9Cr2WVTa, the impurities have small effects.

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5.1 SIMULATING THE PRODUCTION OF FREE DEFECTS IN IRRADIATED METALS – H. L. Heinisch (Pacific Northwest Laboratory).....	125

Under cascade-producing irradiation by high energy neutrons or charged particles, only a small fraction of the initially displaced atoms contribute to the population of free defects, i.e. those that are available to migrate throughout the metal and cause microstructural changes. Although, in principle, computer simulations of free defect production could best be done using molecular dynamics, in practice, the wide ranges of time and distance scales involved can be done only by a combination of atomistic models that employ various levels of approximation. An atomic-scale, multimodel approach has been developed that combines molecular dynamics, binary collision models and stochastic annealing simulation. The annealing simulation is utilized in calibrating binary collision simulations to the results of molecular dynamics calculations, as well as to model the subsequent migration of the defects on more macroscopic time and size scales. The annealing simulation and the method of calibrating the multimodel approach are discussed, and the results of simulations of cascades in copper are presented. The temperature dependence of free defect production following simulated annealing of isolated cascades in copper shows a differential in the fractions of free vacancies and interstitial defects escaping from the cascade above Stage V. This differential, a consequence of the direct formation of interstitial clusters in cascades and the relative thermal stability of vacancy and interstitial clusters during subsequent annealing, is the basis for the production bias mechanism of void swelling.

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Four martensitic steels were examined by transmission electron microscopy after irradiation in the Fast Flux Test Facility (FFTF). Irradiation in FFTF was at 420°C to about 7.8×10^{26} n/m² (E>0.1 MeV), which gave a displacement damage of about 35 dpa. The steels were those of interest for fusion applications and included two commercial steels, 9Cr-1MoVNb (modified 9Cr-1Mo) and 12Cr-1MoVW (Sandvik HT9), and two experimental reduced-activation steels, 9Cr-2WV and 9Cr-2WVTa. Before irradiation, the tempered martensite microstructures of the four steels contained a high dislocation density, and the major precipitate was M₂₃C₆ carbide, with lesser amounts of MC carbide. Irradiation caused only small changes in these precipitates. Voids were found in all irradiated specimens, but swelling remained below 1%, with the 9Cr-1MoVNb having the highest void density. Although the 12Cr-1MoVW steel showed the best swelling resistance, it also contained the highest density of radiation-induced new phases, which were identified as chi-phase and possibly α'. Radiation-induced chi phase was also observed in the 9Cr-1MoVNb steel. The two reduced-activation steels showed very stable behavior under irradiation: a high density of

dislocation loops (average diameter of 50 nm) replaced the original high dislocation density; moderate void swelling occurred, but no new phases formed. The differences in microstructural evolution of the steels can explain some of the mechanical properties observations made in these steels.

- 6.1.2 ON THE ROLE OF STRAIN RATE, SIZE, AND NOTCH ACUITY ON TOUGHNESS:
A COMPARISON OF TWO MARTENSITIC STAINLESS STEELS – G. E. Lucas,
G. R. Odette, J. W. Sheckherd, K. Edsinger, and B. Wirth (University of California
Santa Barbara). 147

The fracture resistance and micromechanisms of two tempered martensitic steels were characterized over a range of temperatures by both mechanical testing and quantitative fractography. Both HT-9 and F82H undergo a fracture mode transition from quasi-cleavage at low temperature to microvoid coalescence at high temperature. The transition in HT9 is rather gradual, and the transition in F82H is extremely abrupt. While the toughness of F82H was higher in all cases, differences between the two steels depended on test type, strain rate and temperature. In general, F82H had only slightly better properties in the quasi-cleavage regime. For example, the transition temperatures indexed at fracture toughness levels of 100 MPa√m were only 25 and 14°C lower in F82H than HT-9 under static and dynamic conditions, respectively, and they were identical at the 10 J level for Charpy impact tests. However, the fracture resistance of F82H was higher than HT9 in the ductile fracture regime. The Charpy V-notch upper shelf energies reflect complex and extrinsically mediated crack tearing processes that have, at best, very limited fundamental or structural significance. Thus, using an energy of 41J, widely perceived to be appropriate indexing a ductile-to brittle transition temperature (DBTT) for Charpy data, results in a much lower (by 81°C) putative DBTT for F82H compared to HT-9. These results not only demonstrate the inherent non-uniqueness of the so called DBTT, but also call into question the use of Charpy data for ranking the relative performance of various alloys. The similarities in quasi-cleavage fracture and differences in ductile fracture are manifested in the fracture surfaces and are interpreted in terms of the underlying mechanisms and microstructures.

- 6.1.3 IRRADIATION CREEP AND SWELLING OF TWO LMR HEATS OF HT9 –
F. A. Garner (Pacific Northwest Laboratory) and M. B. Toloczko (University of
California at Santa Barbara). 169

The irradiation creep and void swelling of two LMR heats of HT9 are analyzed after irradiation at ~400, 495, 550, and 600°C to damage levels ranging from 60 to 174 dpa. Void swelling ceases somewhere between 400 and 495°C. Swelling appears to be somewhat stress-sensitive, however, increasing with stress level. When compared with earlier results on another fusion heat of HT-9, both the swelling and creep appear to be somewhat variable from heat-to-heat. The variability of creep appears to arise from the swelling-enhanced creep component.

At higher temperatures there appears to arise a stress-activated component of strain that increases with temperature. This strain component eventually dominates the strain behavior at the highest temperatures and is probably caused by a combination of stress-activated phase changes and radiation-enhanced primary creep.

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NO CONTRIBUTIONS.

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- 6.3.1 CHARACTERIZATION OF V-4Cr-4Ti HEAT 832665 – M. L. Grossbeck, D. J. Alexander, J. J. Henry, Jr., W. S. Eatherly, and L. T. Gibson (Oak Ridge National Laboratory). 183

A new 500 Kg heat of V-4Cr-4Ti (Heat 832665) is now being characterized by various national laboratories. The Oak Ridge National Laboratory (ORNL) has received sheet of several thicknesses from Argonne National Laboratory (ANL); characterization by chemical analysis, Charpy impact testing, and metallography is in progress. The Charpy tests have shown that the material does not experience a ductile to brittle transition temperature (DBTT) even at temperatures as low as -196°C .

- 6.3.2 RECOVERY AND RECRYSTALLIZATION STUDY ON VANADIUM ALLOYS – A. N. Gubbi, A. F. Rowcliffe, and W. S. Eatherly (Oak Ridge National Laboratory). 187

A series of vacuum-anneals at temperatures from 900° to 1100°C for 1 to 4 h was carried out on vanadium alloys with Cr and Ti contents ranging from 3 to 6 wt.%. Compositional variants of vanadium alloys (~15-kg melt) and a large heat (~500-kg melt) of V-4Cr-4Ti alloy were studied in this work. Optical microscopy, TEM, and microhardness testing were carried out. The alloys tested followed the metallurgically well-established axiom that longer times at low temperatures and shorter times at high temperatures were needed for complete recrystallization. The recrystallization kinetics was faster in the alloys with higher amount of cold work compared to that exhibited by alloys with lower cold work. The large heat of V-4Cr-4Ti alloy with 40% CW showed recovery for anneals at 900°C , and began recrystallizing at 950°C . Complete recrystallization in this alloy occurred at 1000°C , with grain growth for temperatures of 1050°C and above. Recovery and recrystallization kinetics were faster for the small heats because of the higher level of cold work (49%) in the starting material. However, variations in Cr and Ti over the range 3 to 6 wt % had no discernible effect on recovery/recrystallization behavior. The hardness of both recovered and recrystallized structures increased with total (Cr + Ti) content.

- 6.3.3 IMPACT TESTING AND FRACTURE BEHAVIOR OF VANADIUM ALLOYS – A. N. Gubbi, A. F. Rowcliffe, D. J. Alexander, M. L. Grossbeck, and W. S. Eatherly (Oak Ridge National Laboratory). 203

Charpy impact testing was completed on vanadium alloys with Cr and Ti contents ranging from 3 to 6 wt.%. A large heat (~500-kg melt) of V-4Cr-4Ti (heat 832665) and small heats (~15-kg melt each) of compositional variants, V-3Cr-3Ti, V-4Cr-4Ti-Si, V-5Cr-5Ti, V-6Cr-3Ti, and V-6Cr-6Ti, were examined in this work. One-third-size Charpy impact specimens, machined from 3.81-mm-thick plates of these vanadium alloys, were used for impact testing. In a fully recrystallized condition with a grain size of $\sim 16\ \mu\text{m}$, the large heat of V-4Cr-4Ti exhibited a high level of resistance to cleavage failure with a DBTT at $\sim -190^{\circ}\text{C}$. The small (15 kg) heat of V-4Cr-4Ti heat treated to produce the same microstructural condition exhibited similar Charpy impact properties. The small heats containing higher concentrations of Cr and Ti, in a fully recrystallized condition exhibited a DBTT at around -100°C , whereas the V-3Cr-3Ti alloy failed by pure ductile shear at liquid nitrogen temperatures.

- 6.3.4 EFFECT OF HEAT TREATMENT AND TEST METHOD ON DBTT OF A V-5Cr-5Ti ALLOY – Hauxin Li (Associated Western Universities-Northwest Division), M. L. Hamilton and R. H. Jones (Pacific Northwest Laboratory). 215

Specimens annealed at 1125°C for 1 h and furnace cooled were brittle at room temperature (RT) and experienced a mixture of intergranular and cleavage fracture. Fracture toughness (J_{IQ}) at RT was $52\ \text{kJ/m}^2$ and the Charpy-V impact fracture energy (IFE) on one-third scaled specimens was 0.2 J. While material exhibited high fracture toughness at 100°C (J_{IQ} was $485\ \text{kJ/m}^2$) and did not fracture during an impact test, the fracture surface contained a mixture of dimple and intergranular fracture, with intergranular fracture making up to 40% of the total

fracture surface. The ductile to brittle transition temperature (DBTT) was estimated to be above RT from the IFE vs. temperature curve. When material was given an additional annealing at 890°C for 24 h, it became ductile at RT and fractured by microvoid coalescence. The J_{IQ} value increased from 52 kJ/m² to ≈1100 kJ/m². During impact tests, the specimens did not fracture at -100°C and warmer due to a large amount of plastic deformation. The DBTT was -100°C. However, when evaluated by J-integral testing, the material became brittle at -50°C and fractured by cleavage, yielding a J_{IQ} value of 50 kJ/m². The DBTT_y estimated from J_{IQ} vs. temperature was above -50°C, 50°C higher than that from the IFE vs. temperature curve. The result indicated that the V-5Cr-5Ti alloy was sensitive to crack 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, more second phase particles were found in the specimens annealed at 1125°C plus 890°C. Energy dispersive x-ray spectroscopy analysis of the particles indicated that they contained higher Ti concentration. The results indicated that the improved toughness of the specimens annealed at 1125°C plus 890°C probably resulted from the reduced S concentration on the grain boundaries and precipitation of the second phases. It was found that the embrittlement was thermodynamically reversible because the embrittlement could be restored by giving the ductile material additional annealing at 1125°C for 1 h.

- 6.3.5 EFFECT OF PREIRRADIATION HEAT TREATMENT ON SWELLING OF NEUTRON-IRRADIATED VANADIUM-BASE ALLOYS – B. Loomis, L. J. Nowicki, and D. L. Smith (Argonne National Laboratory). 237

The dependence of swelling of neutron-irradiated V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-0.3Si, and V-18Ti alloys on preirradiation heat treatment (which consisted of a 1-h annealing at either 850, 950, 1100, 1125, or 1200°C) was determined from density measurements of the alloys after irradiation at either 420, 520, or 600°C to 21-88 dpa. Swelling of the V-14Cr-5Ti alloy was minimal after 1-h annealing at 1125°C, whereas swelling of the V-7Cr-15Ti alloy was minimal after 1-h annealing at 1125 and 1200°C. Swelling of the V-3Ti-0.3Si alloy increased when the annealing temperature was increased from 850°C to 1200°C, and swelling of the V-18Ti alloy was minimal after preirradiation annealing at 1125°C.

- 6.3.6 MICROSTRUCTURAL EVOLUTION OF V-4Cr-4Ti DURING DUAL-ION IRRADIATION AT 350°C – J. Gazda and M. Meshii (Northwestern University), and B. A. Loomis and H. M. Chung (Argonne National Laboratory). 243

The preliminary results of TEM investigation of microstructural evolution of V-4Cr-4Ti (Heat #832665) alloy irradiated with 4.5 MeV ⁵⁸Ni⁺⁺ ions at 350°C, with and without simultaneous ³He⁺ injection, are presented. This work is the basis of an extensive study designed to evaluate ion irradiation experiments as a tool for simulating and understanding fusion neutron damage and helium generation in V-Cr-Ti alloys. The effects of ion-irradiation damage (at moderate temperatures of <400°C) on mechanical properties of these alloys will be also evaluated in this study. This initial report includes descriptions of specimen preparation techniques, procedures performed during ion irradiation, postirradiation analysis, and results of preliminary transmission electron microscopy (TEM) investigation. Specimens irradiated to ≈10 dpa by ⁵⁸Ni⁺⁺ ions showed a high density of "black-dot" defects and dislocations. Cavity formation in the specimens irradiated simultaneously with ³He⁺ ions to a rate of ≈5 appm/dpa He was not observed.

- 6.3.7 EFFECT OF OXIDATION ON TENSILE BEHAVIOR OF V-5Cr-5Ti ALLOY – K. Natesan and W. K. Soppet (Argonne National Laboratory). 247

Oxidation studies were conducted on V-5Cr-5Ti alloy specimens at 500°C in air to evaluate oxygen uptake of the alloy as a function of temperature and exposure time. The oxidation rates derived from thermogravimetric testing are 5, 17, and 27 μm after one year of exposure

at 300, 400, and 500°C, respectively. Uniaxial tensile tests were conducted on preoxidized specimens of the alloy to examine the effects of oxidation and oxygen migration on tensile strength and ductility. Microstructural characteristics of several of the tested specimens were characterized by electron optic techniques. Correlations have been developed between tensile strength and ductility. Microstructural characteristics of several of the tested specimens were characterized by electron optic techniques. Correlations have been developed between tensile strength and ductility of the oxidized alloy and microstructural characteristics such as oxide thickness, depths of hardened layers, depths of intergranular fracture zones, and lengths of transverse cracks.

6.3.8 IMPACT PROPERTIES OF PRECRACKED V-4Cr-4Ti CHARPY SPECIMENS – H. M. Chung, L. Nowicki, and D. L. Smith (Argonne National Laboratory). 253

Laboratory- and production-scale (20 and 500 kg, respectively) heats of V-4Cr-4Ti, which is the reference vanadium alloy for application in fusion reactor structural components, have recently been produced successfully. Charpy tests conducted previously at -196 to 200°C on 1/3 size blunt-notch specimens, showed that both heats have excellent impact properties, i.e., ductile-brittle transition temperature (DBTT) lower than -200°C and upper-shelf energy of 10-16 J. Effects of precracking on the impact behavior of the Charpy specimens were investigated in this study. Precracked specimens were tested after annealing of the optimal conditions of 1000°C for 1 h. Precracked specimens from both the laboratory-and production-scale heats exhibited normalized energies of 6.7 to 10.9 J at test temperatures of -196 to 200°C; no brittle fracture was observed. This demonstrates the excellent dynamic toughness of V-4Cr-4Ti.

6.3.9 FABRICATION AND IMPACT PROPERTIES OF LABORATORY-SCALE HEAT OF V-5Cr-5Ti – H. M. Chung, L. Nowicki, D. Busch, and D. L. Smith (Argonne National Laboratory). 259

Impact properties were determined on a new 15-kg laboratory heat of V-5Cr-5Ti, fabricated by the same procedures as those used to produce a 500-kg production-scale heat of V-4Cr-4Ti, to identify an optimal annealing procedure for the alloy. Charpy-impact tests were conducted on one-third-size specimens because low-temperature (<0°C) impact properties have been known to be most sensitive to the structure and toughness of the V-(4-5)Cr-(4-5)Ti alloy class. After final annealing at ≈1000°C for 1 h in a high-quality vacuum, the laboratory heat of V-5Cr-5Ti exhibited impact properties as excellent as those of the production- and laboratory-scale heats of V-4Cr-4Ti; i.e., ductile-brittle-transition temperatures less than -200°C and absorbed energies of 10-16 J. This finding demonstrates that, when fabricated by the procedure specified in this study and annealed at the common optimal condition of 1000°C for 1 h, the V-(4-5)Cr-(4-5)Ti alloy class exhibits excellent impact toughness and a sufficient tolerance to minor variations in alloying-element composition.

6.3.10 TENSILE PROPERTIES OF UNIRRADIATED V-Cr-Ti ALLOYS, AND ALTERNATIVE APPROACHES FOR STRENGTHENING THE V-4Cr-4Ti ALLOY – B. A. Loomis, L. J. Nowicki, and D. L. Smith (Argonne National Laboratory). 265

The temperature dependence of tensile properties of unirradiated V-Cr-Ti alloys are presented in the form of tables and figures in this report. These tensile-property data, together with other physical and mechanical property data for unirradiated and neutron-irradiated V-Cr-Ti alloys, are examined for alternative approaches to strengthen the V4Cr-4Ti alloy, which is the current prime-candidate vanadium-base alloy for use as structural material in a fusion reactor. Consideration of three alternative approaches for strengthening (i.e., increased Cr and/or Ti concentration; heat treatment; or increased Si and/or Si, Al, and Y concentration) lead us to recommend Si and/or Si, Al, and Y additions as most promising for strengthening of the V4Cr-4Ti alloy without major impact on the physical and mechanical properties of V-4Cr-4Ti.

- 6.3.11 EFFECT OF ANNEALING ON IMPACT PROPERTIES OF PRODUCTION-SCALE
EAT OF V-4Cr-4Ti – H. M. Chung, L. Nowicki, and D. L. Smith (Argonne National
Laboratory). 273

A 500-kg heat of V-4Cr-4Ti, an alloy identified previously as the primary vanadium-based candidate alloy for application in fusion reactor structural components, has been successfully produced. Impact tests were conducted at -196 to 150°C on one-third-size blunt-notch Charpy specimens of the scaleup heat in as-rolled condition and after annealing for 1 h at 950, 1000, and 1050°C in a high-quality vacuum. The annealed material remained ductile at all test temperatures; the ductile-brittle transition temperature was lower than -200°C. The upper-shelf energy of the production-scale heat was similar to that of the laboratory-scale (\approx 30-kg) heat of V-4Cr-4Ti investigated previously. The effect of annealing temperature between 950 and 1050°C was not significant; however, annealing at 1000°C for 1 h not only produced the best impact properties but also ensured a sufficient tolerance to the effect of temperature inhomogeneity that is expected when large components are annealed. The effect of the notch geometry of the Charpy-impact specimens was also investigated. When annealed properly (e.g., at 1000°C for 1 h), impact properties were not sensitive to notch geometry (45°-notch, root radius 0.25 mm; and 30°-notch, root radius 0.08 mm).

- 6.4 COPPER ALLOYS. 279

- 6.4.1 HIGH TEMPERATURE STABILITY OF DISPERSION STRENGTHENED COPPER
ALLOYS IRRADIATED WITH FAST NEUTRONS – D. J. Edward (Associated Western
Universities, F. A. Garner and M. L. Hamilton (Pacific Northwest National Laboratory),
and J. D. Troxell (SCM Metal Products, Inc.). 281

Two dispersion strengthened copper alloys, GlidCop CuAl25 and GlidCop-Nb, were irradiated under three different conditions to study their response to high temperature neutron irradiation. Previous studies demonstrated that GlidCop CuAl25 experienced a decrease in yield and ultimate strength by 50 dpa, but no further changes in strength occurred at doses up to 150 dpa. The implications of this are that cold worked (CW) CuAl25 alloys will experience most of the changes in mechanical properties at dose levels that are well within the Basic Physics Phase of ITER's operation. Alloying CuAl25 with 10 wt% Nb produced a DS copper alloy that was completely resistant to any changes in strength during irradiation. These results, when combined with earlier studies, strongly suggest that high temperature neutron irradiation relaxes the dislocation structure within a few dpa (5.8 dpa or less). Alloying with niobium is thought to effectively prevent this relaxation, thereby maintaining the strength of the material.

- 6.4.2 BRAZED DISPERSION STRENGTHENED COPPER: THE EFFECT OF NEUTRON
IRRADIATION AND TRANSMUTATION ON BOND INTEGRITY – D. J. Edwards
(Associated Western Universities), F. A. Garner and M. L. Hamilton (Pacific Northwest
Laboratory), and J. D. Troxell (SCM Metal Products, Inc.). 299

Four types of brazes were used to join sheets of GlidCop CuAl25. Miniature tensile specimens and TEM disks were fabricated from the joints, and irradiated under various conditions to study their response to high temperature neutron irradiation. Two of the brazes, TiCuAg and TiCuNi, were eliminated from consideration because of the poor quality of the brazed joints. Brazed joints produced using a gold-containing braze were satisfactory for the unirradiated state. However, transmutation of Au to Hg affected the integrity of some of the joints in the specimens irradiated in a below-core position where the neutron spectrum was much softer. A CuAg braze yielded satisfactory joints in the unirradiated state, and held up very well when the irradiated specimens were tested. However, transmutation of Ag to Cd leads to a high residual radioactivity that limits the usefulness of this braze after exposure to neutron irradiation. Further work is necessary to

identify brazes that are "transmutation-resistant" and also that minimize the potential for activation by a suitable choice of elemental constituents.

- 6.4.3 ROOM TEMPERATURE FATIGUE BEHAVIOR OF CuCrZr OF TWO SIZES – K. Leedy and J. F. Stubbins (University of Illinois), F. A. Garner and D. J. Edwards (Pacific Northwest Laboratory), and B. N. Singh (Risø National Laboratory). 315

The room temperature fatigue behavior of unirradiated CuCrZr in two specimen sizes has been measured. The fatigue performance was found to be intermediate to those of OFHC copper and CuAl25, which were reported earlier. The size effects correlation is complicated somewhat by the sensitivity of this alloy to details of the heat treatment. Declining interest in this alloy for ITER applications and the shut-down of EBR-II will probably preclude irradiation of this alloy.

- 6.5 ENVIRONMENTAL EFFECTS IN STRUCTURAL MATERIALS. 319

- 6.5.1 FABRICATION OF ALUMINUM NITRIDES AND ITS STABILITY IN LIQUID ALKALI METALS – K. Nateson and D. L. Rink (Argonne National Laboratory). 321

Aluminum nitride (AlN) has been selected as a prime candidate to electrically insulate the V-alloy first wall in the self-cooled concept for ITER application. Detailed investigations were conducted on the fabrication, metallurgical microstructure, compatibility in liquid Li, and electrical characteristics of AlN material obtained from several sources. Coating fabrication methods included physical vapor deposition, reaction sputtering, ion-beam-assisted deposition, chemical vapor deposition, and a chemical route. Microstructural characterization of the coated samples was conducted by scanning electron microscopy, energy-dispersive X-ray analysis, and X-ray diffraction. Lithium compatibility studies were conducted in static systems by exposure of AlN-coated specimens to Li for several time periods. Electrical resistance measurements were made at room temperature on the specimens before and after exposure to liquid Li. The results obtained in this study indicate that AlN is a viable coating from the standpoint of chemical compatibility in Li, electrical insulation characteristics, and ease of fabrication, and that the coating should be examined further for fusion reactor application.

- 6.5.2 ELECTRICAL INSULATOR COATINGS ON V ALLOYS – J.-H. Park, G. Dragel, and W. D. Cho (Argonne National Laboratory). 329

Several intermetallic films were applied to V alloys to provide electrical insulation and corrosion resistance. Grain-growth behavior for the V-5Cr-5Ti alloy at 1000°C was investigated to determine the stability of the alloy substrate during coating formation by chemical vapor deposition or metallic vapor processes at 800-850°C. Film layers were examined by optical and scanning electron microscopy and by electron-energy-dispersive and X-ray diffraction analysis; they were also tested for electrical resistivity and corrosion resistance. The results elucidated the nature of the coatings, which provided both electrical insulation and high-temperature corrosion protection.

- 6.5.3 FORMATION AND SELF-HEALING BEHAVIOR OF CaO INSULATOR COATINGS ON A VANADIUM-BASE ALLOY IN LIQUID LITHIUM – J.-H. Park, and T. F. Kassner (Argonne National Laboratory). 339

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 $\approx 10^6 \Omega$ at 400°C. Thermal cycling between 300

and 700°C changed the resistance of the coating layer, which followed insulator behavior. Examination of the specimens after cooling to room temperature revealed no spallation, but homogeneous crazing cracks were present in the CaO coating. In-situ self-healing of the cracks occurred at temperatures $\geq 360^\circ\text{C}$. These results suggest that thin coatings can be produced on variously shaped surfaces by controlling the exposure time, temperature, and composition of the liquid metal.

6.5.4 SELECTION OF A LIQUID CALCIUM-LITHIUM ALLOY FOR FABRICATING CaO INSULATOR COATINGS ON V-5%Cr-5%Ti – J.-H. Park and T. F. Kassner (Argonne National Laboratory). 347

An electrically insulating coating at the liquid-metal/structural-material interface of a magnetic fusion reactor is required to prevent adverse currents generated by the magnetohydrodynamic (MHD) force from passing through the structural walls. Thin, homogeneous, electrically insulating CaO coatings can be produced on variously shaped surfaces of V-5%Cr-5%Ti by exposing the alloy at controlled times and temperatures to liquid Li containing Ca. Formation of Ca-Li alloys by dissolution of solid Ca in liquid Li in an Ar environment was investigated, and the exothermic heat of solution was found to be low, as indicated by minimal increases in temperature. The recommended composition of a liquid alloy for fabrication of this coating on reactor components is Li-82 wt.% Ca (44 at.% Ca), which has a liquidus temperature of $\approx 230^\circ\text{C}$. As the solute Ca in the liquid alloy is consumed during the coating process, the liquidus temperature of the alloy decreases. This coating 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 at $\approx 400^\circ\text{C}$. Cracks that form in the CaO coating during thermal cycling exhibit in-situ self-healing behavior at temperatures $\geq 360^\circ\text{C}$.

7.0 SOLID BREEDING MATERIALS AND BERYLLIUM. 353

7.1 INVESTIGATION OF TRITIUM RELEASE FROM TITANATE – J. P. Kopasz and C. E. Johnson (Argonne National Laboratory). 355

Tritium release from lithium titanate has been investigated using isothermal anneal experiments. These experiments suggest that the rate-controlling step for tritium desorption is desorption of HTO from a titanium site on the Li_2TiO_3 surface. The experiments also indicate that the presence of hydrogen in the purge gas helps the tritium release by increasing the fraction of tritium released at a given temperature.

7.2 *Ab initio* CALCULATIONS FOR DISSOCIATIVE HYDROGEN ADSORPTION ON LITHIUM OXIDE SURFACES – A. Sutjianto, S. W. Tam, L. Curtiss, and C. E. Johnson (Argonne National Laboratory) and R. Pandey (Michigan Technological University). 359

Dissociative hydrogen chemisorption on the Li_2O surfaces of the (100), (110), and (111) planes has been investigated with *ab initio* Hartree-Fock calculations. Calculations for unrelaxed crystal Li_2O structures indicated that except for the (100) surface, the (110) and (111) surfaces are stable. Results on the heterolytic sites of n-layer (110) slabs (where $n \geq 2$) and three-layer (111) slabs suggest that dissociative hydrogen chemisorption is endothermic. For a one-layer (110) slab at 100% surface coverage, the dissociative hydrogen chemisorption is exothermic, forming OH^- and $\text{Li}^+\text{H}^-\text{Li}^+$. The results also indicate that the low coordination environment in surface step structure, such as kinks and ledges, may play an important role in the hydrogen chemisorption process. On the homolytic sites of the (110) and (111) surfaces, there is no hydrogen chemisorption.

8.0 CERAMICS 369

- 8.1 FATIGUE BEHAVIOR OF NICALON/SiC COMPOSITES – N. Miriyala, P. K. Liaw, and C. J. McHargue (University of Tennessee), X. Mao and W. Mao (University of Calgary), L. L. Snead (Oak Ridge National Laboratory), and D. K. Hsu (Iowa State University). 371

A periodic model using a finite element method (FEM) was developed to predict the effect of porosity on the in-plane and through-thickness elastic stiffness constants of Nicalon/SiC composites. The FEM results indicated that the in-plane moduli values will be higher than the through-thickness moduli values. Also, the predicted values were in close agreement with the ultrasonically measured elastic stiffness constants for the composite materials under study.

- 8.2 MICROSTRUCTURE OF AL₂O₃ IRRADIATED WITH AN APPLIED ELECTRIC FIELD – S. J. Zinkle, J. D. Hunn, and R. E. Stoller (Oak Ridge National Laboratory). 379

A thin amorphous film of alumina was irradiated with 2-MeV He⁺ ions at ~400°C up to a damage level of about 0.01 displacements per atom (dpa). The alumina films were sufficiently thin (~1.8 μm) to allow the ion beam to be completely transmitted through the specimen. An electric field of ~280 V/mm (dc) was applied continuously during the irradiation. Radiation induced electrical degradation (RIED), i.e. a permanent increase in the conductance of the film, was observed in specimens irradiated at temperatures near 400 to 450°C, but did not occur in a specimen irradiated above 500°C. An investigation by transmission electron microscopy found no evidence for colloid formation. The observed increase in the conductance of the alumina film may be due to radiation--induced microcracking.

- 8.3 INVESTIGATION OF RADIATION INDUCED ELECTRICAL DEGRADATION IN ALUMINA UNDER ITER-RELEVANT CONDITIONS – L. L. Snead, D. P. White, and S. J. Zinkle (Oak Ridge National Laboratory). 385

An in-situ experiment investigating the radiation induced electrical degradation (RIED) effect in polycrystalline alumina is described. A Wesgo AL-995 polycrystalline alumina sample has been irradiated with fission neutrons to 1.4 displacements per atom (dpa) at 340-365°C with no evidence of RIED. The implication of these and previous results are discussed in terms of their impact on the International Thermonuclear Experimental Reactor (ITER), with the conclusion that RIED will be of no consequence during the basic phase of the machines operation.

- 8.4 X-RAY-INDUCED LUMINESCENCE FROM HYDROXYL-DOPED SILICA FIBERS -- D. W. Cooke, E. H. Farnum, F. W. Clinard, Jr., B. L. Bennett (Los Alamos National Laboratory) and A. M. Portis (UC-Berkeley). 397

Fiber optics is expected to play a very important role in ITER diagnostics. The present consensus is that silica fibers comprised of low-OH silica cores and F-doped cladding are the best candidates for use in optical diagnostic systems in a high-fluence radiation environment. One concern regarding their use, however, is radiation-induced visible luminescence. Accordingly, we have examined the luminescence of low- (fiber A) and high-OH (fiber S) content silica fibers subjected to continuous x irradiation as a function of temperature and time. In the interval 7 - 300 K, fiber A exhibits two well-defined peaks at 520 and 670 nm with the exact position being dependent upon temperature. Fiber S is characterized by one broad peak near 590 nm with evidence of a second weak peak near 620 nm. Generally we find that luminescence peak intensities decay with increasing temperature. The time dependence of each peak was measured at various fixed temperatures and was found to exhibit exponential decay. Rate equations were written to explain the time dependent intensities in terms of

excitation and luminescence center creation and annihilation. Results suggest that Cherenkov radiation may be more important than luminescence in diagnostic fibers.

- 8.5 OXYGEN EFFECTS ON SiC/SiC COMPOSITES FOR FUSION STRUCTURAL APPLICATIONS – G. D. Springer, C. F. Windisch, Jr., C. H. Henager, Jr., and R. H. Jones (Pacific Northwest Laboratory). 407

Linear-parabolic kinetics governed the oxidation reaction with 100 to 1500 ppm oxygen at 1100°C. This behavior concurs with the previous research of Windisch et al., which observed a deviation from linearity beginning below 2500 ppm. By focusing on the linear region, a simple model estimated the linear rate dependency on oxygen partial pressure to be on the order of 0.911. Future tests will be performed with 100 and 1000 ppm in the temperature range of 800 to 1000°C in order to refine the understanding of temperature and partial pressure effects. The relationship between interfacial oxidation and experimental parameters, such as temperature, pressure, and interfacial thickness, will be described in an upcoming comprehensive model.

- 8.6 HYDROGEN EFFECTS ON SiC/SiC COMPOSITES FOR FUSION STRUCTURAL APPLICATIONS – G. D. Springer and R. H. Jones (Pacific Northwest Laboratory). 413

Two exploratory experiments at 1100°C and 1200°C in an argon +1% hydrogen environment have demonstrated a relatively slow reaction rate. This slow reaction rate concurs with literature, yet a better understanding of kinetics is still needed. Further experimentation will test the reproducibility of these early results and better determine the influence of experimental conditions by varying temperature and the partial pressure of hydrogen.

- 8.7 DIMENSIONAL STABILITY OF SiC-TYPE FIBERS NEUTRON IRRADIATED TO HIGH DOSES – G. E. Youngblood, D. J. Senor, and G. W. Hollenberg (Pacific Northwest Laboratory). 417

Silicon carbide based fibers with a range of stoichiometries and microstructures (Nicalon CG, Nicalon HVR, HPZ, Tyranno, and Dow/NASA Xstalline) were selected for evaluation of their dimensional stability after neutron irradiation to high doses. For comparison, carbon fibers with a range of graphitization also were evaluated.

The fibers were irradiated in the MOTA 2B cycle of the FFTF reactor. Two sets of the selected C and SiC fiber types were exposed at 430°C to a fluence of 5.5×10^{21} n/cm² or 2.5×10^{22} n/cm² ($E \geq 0.1$ MeV), equivalent to relatively high doses of 5.3 and 25 dpa-SiC. Dimensional stability was determined by measuring the length and density changes of the fibers after the irradiations.

For the SiC-based fibers above 5 dpa-SiC, little dose dependence was observed except for the Tyranno fiber. The HPZ, Tyranno, and Nicalon HVR fibers, whose pre-irradiated densities were quite low, exhibited substantially more axial shrinkage than the Nicalon CG fiber. Even though the shrinkage of the Nicalon CG fiber was moderate (about 2% at 430°C), fiber shrinkage and debonding from the matrix previously had been observed to result in decreased strengths in Nicalon fiber SiC/SiC composites irradiated to the same fluence and at similar and at higher temperatures. The developmental fiber, Dow/NASA Xstalline, actually exhibited slight swelling rather than shrinkage. The composition of the Dow/NASA fiber was near stoichiometric SiC, the density was 90% of theoretical for SiC, and the microstructure was reported to be more crystalline than for the other SiC-based fibers.

For the C fibers, the amount of axial shrinkage was much greater than observed for the irradiated SiC-based fibers and generally was greater the lower the degree of initial graphitization. Also, in contrast to the irradiated SiC behavior, the radiation damage did not

appear to saturate, but continuously increased with increasing dose. The axial shrinkage exceeded about 20% for the most graphitic C fiber and exceeded 60% for the less graphitic fiber at the higher dose. Independent of the initial degree of graphitization, the densities of all the C fibers increased and appeared to saturate at a common value by the 3 dpa-C dose. With increasing dose above 3 dpa-C, the densities increased and returned almost to their unirradiated values by 15 dpa-C. The density change data indicated that C fibers will continuously shrink in the axial direction (i.e., the preferred alignment of the graphitic a-axis in C fibers) under irradiation. However, in the diametral direction, graphitic c-axis growth will be accommodated by porosity and/or amorphicity initially, but with continued irradiation diametral swelling will commence once the accommodation is over.

Silicon carbide based fibers that are close to stoichiometric and crystalline appear to have the radiation damage tolerance necessary to make SiC/SiC composites suitable for further testing and development for fusion power system applications. Due to the fundamental nature of radiation damage in graphite, dimensional instability (extreme axial shrinkage and diametral swelling) of C fiber irradiated to high doses will prevent their use in C/C composites for long-term operations in a fusion power system.