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- 1.1 A GENERALIZED CONSTITUTIVE MODEL FOR A V-4Cr-4Ti ALLOY –**
 E. Donahue, G. R. Odette, G. E. Lucas (University of California) 3

A physically based constitutive model for low-to-intermediate temperatures, strains and strain rates is derived for the program heat of the V-4Cr-4Ti. The supporting database is based on tensile tests carried out over a wide range of temperatures and strain rates. The overall constitutive model is based on additive yield and post yield strain hardening contributions to the flow stress. The yield stress has both thermally activated and athermal components. The former can be described by a two-mechanism activated dislocation slip model, with contributions from both lattice friction (lower temperature) and interstitial obstacles (higher temperature). The model uses a weighted average of the two mechanisms as a function of a strain rate compensated temperature. Post-yield strain hardening was found to be essentially athermal. Strain hardening can be fit by a two-component modified Voce-type model, which predicts saturating hardening behavior. The constitutive model is used to determine flow stability limits as estimates of uniform tensile strains. The relatively compact, but mechanism-based, model has a number of both fundamental and practical advantages that are briefly outlined. Extensions to directly model compositionally and microstructurally mediated mechanisms, including the effects of irradiation, and key phenomena, such as flow localization, are important objectives of future research.

- 1.2 ON THE MECHANISMS AND MECHANICS OF FRACTURE IN A**
V-4CR-4TI ALLOY: MODELING THE EFFECTS OF IRRADIATION,
LOADING RATE AND SPECIMEN CONFGURATION ON TOUGHNESS-
TEMPERATURE CURVES – E.G. Donahue, G.R. Odette, G.E. Lucas
 (University of California, Santa Barbara) 9

The fracture behavior of the 500 kg heat of the V-4Cr-4Ti was used to characterize and model of low dose, low-to-intermediate temperature irradiation, loading rate and specimen configuration on toughness-temperature curves. A critical stress–critical stressed area cleavage criteria predicts effective toughness-temperature curves in the cleavage transition regime, including the influence of size and geometry that mediate the loss of triaxial constraint. It appears that cross rolling lowers the cleavage transition temperature. Irradiation and high loading rates result in significant upward shifts in the transition temperature, due to corresponding increases in the yield stress. A simple equivalent yield stress model accurately predicts these effects. Flow localization following irradiation has a profound effect on effective ductile fracture toughness, qualitatively consistent with effects of irradiation on the tensile properties, dominated by reductions in uniform strain. The large cleavage transition temperature shifts and ductile toughness decreases may limit the application of V-4Cr-4Ti alloys for low-to-intermediate temperature irradiation environments.

- 1.3 UNIAXIAL CREEP BEHAVIOR OF V-4Cr-4Ti ALLOY** – K. Natesan,
W. K. Soppet, and D. L. Rink (Argonne National Laboratory) 17

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.

- 1.4 MICROSTRUCTURAL EXAMINATION ON V-4Cr-4Ti RODS AND CREEP TUBES** – Y. Yan, H. Tsai, and D. L. Smith (Argonne National Laboratory) 23

Extruded V-4Cr-4Ti bar stock from ANL's 500-kg 832665 heat and swaged rod stock from GA's 1000-kg 832864 heat were examined using optical microscopy and transmission electron microscopy to determine whether they are suitable feedstock for the upcoming creep tubing fabrication campaign. To compare microstructure, 832665-heat creep tubing from the last fabrication campaign was also examined. The results of this study show a banded (stringer) structure, consisted of fine Ti-rich particles, exists in both the 832665 and 832864 bars. In the finished creep tubing, remnant of Ti-rich secondary phase particles could also be found although the inhomogeneity is less pronounced than in the feedstock. A homogenization treatment (e.g., annealing at 1200°C for 2 h) may remove the banded structure and this will be tested on small pieces of the 832665 and 832664 bars in the near future.

- 1.5 EFFECT OF OXYGEN ON THE CRACK GROWTH BEHAVIOR OF V-4Cr-4Ti at 600°C** – R. J. Kurtz (Pacific Northwest National Laboratory) 32

Exploratory experiments were performed to evaluate the effect of oxygen on the crack growth response of V-4Cr-4Ti at 600°C under constant load. Tests were run in gettered argon, argon containing 2000 ppm oxygen, and laboratory air using fatigue pre-cracked compact tension specimens. Crack growth was measured primarily by post-test fracture surface examination, but also by in-test compliance measurements. Crack growth rates measured in air and gettered argon were about $2-3 \times 10^{-3}$ mm/h at a stress intensity factor of about 40 MPa \sqrt{m} . The crack growth rate in argon with 2000 ppm oxygen was about 7×10^{-2} mm/h at the same intensity level. The crack growth rates were very sensitive to the stress intensity factor. Over a limited range of stress intensity values the crack growth rate in argon plus 2000 ppm oxygen appears to be power-law dependent on stress intensity with an exponent of about 8.9. The fracture mode in air and gettered argon was transgranular cleavage with 20 to 30% intergranular fracture. In the oxygenated argon environment crack growth occurred predominantly by transgranular cleavage.

- 1.6 OXIDATION OF V-4Cr-4Ti AT LOW PRESSURES** – B. A. Pint,
J. R. DiStefano, J. Bentley, and L. D. Chitwood (Oak Ridge National Laboratory) 40

To complement previous work on V-4Cr-4Ti at 400-500°C, similar oxidation experiments were conducted at 600-700°C. In general, the rates were linear with time. However, at higher oxygen pressures, e.g. 10^{-3} Pa (10^{-5} Torr), specimen surfaces became slightly discolored indicating the formation of a surface oxide and rates were linear-parabolic. The addition of more than 1000 ppm oxygen resulted in

a significant loss of room temperature ductility. Annealing at 950°C was effective in improving ductility at oxygen levels below 1500 ppm. With higher oxygen levels, the anneal was ineffective or further reduced ductility. High resolution analytical electron microscopy was used to examine the oxidized V-4Cr-4Ti microstructure before and after annealing at 950°C. Low ductility before annealing is attributed to fine oxide particles in the matrix inhibiting dislocation movement. Large Ti-rich oxide particles were observed at grain boundaries while the alloy matrix adjacent to the boundary was depleted in Ti.

- 1.7 OXIDATION OF V-4Cr-4Ti ALLOYS CONTAINING Al, Si, and Y –**
M. Fujiwara (Tohoku University) and K. Natesan (Argonne National Laboratory) 45

A systematic study has been conducted to determine the effects of time and temperature on the oxidation behavior of Si-, Al, and Y-modified V-4Cr-4Ti alloys. All samples were from 0.80-mm-thick cold-rolled sheets, and each was annealed in vacuum at 1000°C for 1 h prior to high-temperature exposure. Different samples from each alloy were heated in air between 400 and 620°C for times up to a few hundred hours. Weight change data were used to evaluate the kinetics of oxidation process in the modified alloys, and the rate constants were compared with data developed earlier on base alloy.

- 1.8 STUDY OF THE LONG-TERM STABILITY OF MHD COATINGS FOR FUSION REACTOR APPLICATIONS –** B. A. Pint, L. D. Chitwood, J. H. DeVan, and J. R. DiStefano (Oak Ridge National Laboratory) 49

This project began in June 1999. Initial testing has been performed on AlN + 5% Y₂O₃ at 400-700°C. With increasing temperature, this material was increasingly attacked by lithium in 1000 h tests and there was a corresponding increase in the aluminum content of the lithium. These results suggest that the maximum use temperature of AlN in lithium may be 600°C or less. Currently, 1000 h experiments are being completed with yttria-free AlN and CaO specimens at 400, 500, and 700°C.

- 1.9 ELECTRICAL RESISTIVITY AND MICROHARDNESS MEASUREMENTS OF VANADIUM AND V-4Cr-Ti ALLOY –** D. T. Hoelzer, S. J. Zinkle, and A. F. Rowcliffe (Oak Ridge National Laboratory), and M. K. West (University of Tennessee) 51

The purpose of this study was to investigate the interactions between Ti and interstitial solutes over temperature ranges corresponding to thermally activated processes such as precipitation, dislocation recovery and recrystallization, and grain growth. In this study, room temperature electrical resistivity and microhardness measurements were performed on cold-worked (CW) vanadium, CW V-4Cr-4Ti, annealed V-4Cr-4Ti, and the fusion zone of welded V-4Cr-4Ti plate over the isochronal annealing temperature range from 200 to 1200°C. The results suggested that Ti solutes in the vanadium alloys interacted with interstitial O, C, and N solutes at temperatures of 200°C and higher. Below ~400°C, these interactions influenced processes such as solute diffusivity and dislocation atmosphere formation. Above ~400°C, recovery, recrystallization and precipitation processes had the most significant effect on the property measurements.

- 1.10 RESISTANCE WELDING OF V-4Cr-4Ti ALLOY**– Z. Xu, D. L. Smith, and C. B. Reed (Argonne National Laboratory) 56

More resistance weld samples were prepared on 3.8 mm thick V-4Cr-4Ti alloy plate on a 50 KVA welder with more optimal process parameters. The microstructure of the weld regions were characterized and correlated with the weld parameters. Simplified torque tests were conducted on the test samples to provide a preliminary assessment of the shear strength and ductility of the test welds. Fractography results for the torque samples indicated that the fracture surfaces of resistance welds exhibit ductile characteristics.

- 1.11 HYDROGEN SOLUBILITY IN VANADIUM ALLOYS, LiCa ALLOYS, and SnLi ALLOYS** – D. L. Smith (Argonne National Laboratory), R. E. Buxbaum (REB Research), and C. B. Reed (Argonne National Laboratory) 62

A systematic study is currently being conducted by Argonne National Laboratory with support from REB Research to provide baseline data on the solubility of hydrogen in selected LiCa, SnLi, and VCrTi alloys. The experimental approach involves charging and degassing of vanadium alloys and vanadium alloy encapsulated LiCa and SnLi specimens to various hydrogen partial pressures while maintaining the system at constant temperature. With this procedure one avoids the problems associated with the rapid redistribution of hydrogen that is typically observed during cool-down of the specimens. Initial tests have been conducted on solid cylindrical specimens of a vanadium alloy. The liquid metal alloy will be contained in vanadium alloy capsules. Preliminary results for the V-4Cr-4Ti alloy (Heat #832665) are reported.

- 1.12 STUDY OF IRRADIATION CREEP OF VANADIUM ALLOYS** – H. Tsai, R. V. Strain, M. C. Billone, T. S. Bray, and D. L. Smith (Argonne National Laboratory), M. L. Grossbeck (Oak Ridge National Laboratory), K. Fukumoto and H. Matsui (Tohoku University) 65

Pressurized-tube specimens made from V-4 wt.% Cr-4 wt.% Ti (832665 heat) thin-wall tubing were irradiated in the HFIR RB-12J experiment to study creep under neutron-damage conditions. The calculated dose for the specimens ranged from 5.5 to 6.0 dpa and the calculated irradiation temperatures ranged from 400 to 500°C. The results show the creep rate to be linearly dependent on stress. The 12J data, when combined with the previous ATR-A1 data set, indicate the creep rates could be significant even at moderate stress and dpa levels.

- 1.13 TENSILE PROPERTIES OF V-(Cr,Fe)-Ti ALLOYS AFTER IRRADIATION IN THE HFIR-12J EXPERIMENT** – K. Fukumoto, H. Matsui (IMR/Tohoku University), Y. Yan, H. Tsai, R. V. Strain, and D. L. Smith (Argonne National Laboratory) 70

Postirradiation tensile tests at room temperature and 500°C were performed on V-(Cr,Fe)-Ti alloys specimens irradiated in the HFIR-12J experiment. The specimens were of the SSJ design with overall dimensions of 16.0 × 4.0 × 0.2 mm and gauge dimensions of 5.0 × 1.2 × 0.2 mm. The irradiation temperature was ≈500°C and the attained neutron damage was ≈6 dpa. Results from these tensile tests show all specimens retained respectable elongation and irradiation hardening was modest. The properties of the V-(3-5)Cr-(3-5)Ti alloys appears not to be strongly affected by

the Ti and Cr composition variations. For the V-(3-4)Fe-4Ti-(0-0.1)Si alloys, significantly, the uniform elongation was nearly 10%. The reduction-in-area in all specimens was high, >85%, indicating ductile behavior. These findings show good mechanical properties of V-(Cr,Fe)-Ti alloys after the 500°C neutron irradiation to 6 dpa.

- 1.14 EFFECT OF LOW TEMPERATURE IRRADIATION IN ATR ON THE MECHANICAL PROPERTIES OF TERNARY V-Cr-Ti ALLOYS –**
M. L. Hamilton, M. B. Toloczko, B. M. Oliver, and F. A. Garner (Pacific Northwest National Laboratory) 76
- Tensile tests and shear punch tests were performed on a variety of vanadium alloys that were irradiated in the Advanced Test Reactor (ATR) at temperatures between 200 and 300°C to doses between 3 and 5 dpa. Tests were performed at room temperature and the irradiation temperature. The results of both the tensile tests and the shear punch tests show that following low temperature irradiation, the yield strength increased by a factor of 3-4 while the ultimate strength increased by a factor of approximately 3. Uniform elongation and tensile reduction in area show that the ductility diminishes following irradiation. The correlation between uniaxial ultimate strength and effective shear maximum in strength was in excellent agreement with previous studies on other materials. Using the room temperature test data, the correlation between uniaxial yield strength and effective shear yield strength was in excellent agreement with previous studies on other materials. The yield strength data obtained at the irradiation temperature did not fit the room temperature correlation.
- 1.15 EFFECT OF STRAIN RATE ON THE TENSILE PROPERTIES OF UNIRRADIATED AND IRRADIATED V-4Cr-4Ti –** A. F. Rowcliffe, S. J. Zinkle, and D. T. Hoelzer (Oak Ridge National Laboratory) 96
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- Several rod-shaped specimens with uniaxially packed fibers (Hi-Nicalon, Hi-Nicalon Type S, Tyranno SA, and Amoco K1100 types) and a pre-ceramic polymer matrix have been fabricated. By using appropriate analytic models, the bare fiber thermal conductivity (K_f) and the interface thermal conductance (h) will be determined as a function of temperature up to 1000°C before and after irradiation for samples cut from these rods. Initial results are: (1) for unirradiated Hi-Nicalon SiC fiber, K_f varied from 4.3 up to 5.9 W/mK for the 27-1000°C range, (2) for unirradiated K1100 graphite fiber, K_f varied from 576 down to 242 W/mK for the 27-1000°C range, and (3) $h = 43 \text{ W/cm}^2\text{K}$ at 27°C as a typical fiber/matrix interface conductance.
- 2.2 REACTION-BASED SiC MATERIALS FOR JOINING SILICON CARBIDE COMPOSITES FOR FUSION ENERGY** – C. A. Lewinsohn and R. H. Jones (Pacific Northwest National Laboratory), M. Singh (NASA Glenn Research Center), H. Serizawa (Osaka University), and Y. Katoh and A. Kohyama (Kyoto University) 119
- The fabrication of large or complex silicon carbide-fiber-reinforced silicon carbide (SiC/SiC) components for fusion energy systems requires a method to assemble smaller components that are limited in size by manufacturing constraints. Previous analysis indicates that silicon carbide should be considered as candidate joint materials. Two methods to obtain SiC joints rely on a reaction between silicon and carbon to produce silicon carbide. This report summarizes preliminary mechanical properties of joints formed by these two methods. The methods appear to provide similar mechanical properties. Both the test methods and materials are preliminary in

design and require further optimization. In an effort to determine how the mechanical test data is influenced by the test methodology and specimen size, plans for detailed finite element modeling (FEM) are presented.

- 2.3 EVALUATION OF NEUTRON IRRADIATED NEAR-STOICHIOMETRIC SILICON CARBIDE FIBER COMPOSITES** – L. L. Snead (Oak Ridge National Laboratory), Y. Kato and A. Kohyama (Kyoto University), and J. L. Bailey, N. L. Vaughn, and R. A. Lowden (ORNL) 128

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- 3.1 A UNIFIED MODEL FOR CLEAVAGE TOUGHNESS IN THE TRANSITION** – G. R. Odette (University of California Santa Barbara) 131

A Master Curves-Shifts (MC- ΔT) method has been previously proposed as an engineering expedient to enable the use of small specimens to predict the effects of geometry, irradiation, loading rates on fracture conditions for large fusion structures. However, in addition to other unresolved issues, the MC- ΔT requires a better basic understanding, including the universality of the MC shape. Thus a new unified micromechanics model of fracture toughness in the cleavage transition regime is proposed. The model combines analytical representations of finite element method simulations of crack tip stress fields with a local critical stress-critical stressed area ($\sigma^* \cdot A^*$) fracture criteria. The model, and simpler alternatives, have been very successful in predicting geometry and loading rate effects, as well as irradiation hardening induced Charpy shifts. However, the standard models do not predict a constant MC $K_{Jc}(T)$ shape following irradiation. This apparent inconsistency with experiment is now resolved by incorporating a modest temperature dependence in σ^* that appears to be consistent with an independent body of data. Several experiments suggest high helium levels may increase irradiation induced toughness temperature shifts above levels associated with hardening alone. However, these experiments are all confounded, and must be interpreted with great caution. If real, helium effects may be relatively modest. Single variable experiments and complementary data which will allow a mechanism-based interpretation of the mechanical test data are needed to characterize the influence of helium on fast fracture.

- 3.2 A POTENTIAL FERRITIC/MARTENSITIC STEEL FOR FUSION APPLICATIONS** – R. L. Klueh, N. Hashimoto (Oak Ridge National Laboratory), R. F. Buck (Advanced Steel Technology), and M. A. Sokolov (Oak Ridge National Laboratory) 140

The A-21 steel is a Fe-Cr-Co-Ni-Mo-Ti-C steel that is strengthened by a fine distribution of small titanium carbide (TiC) precipitates formed by thermo-mechanical treatment. After a high-temperature austenitization treatment, the steel is cooled to an intermediate temperature and hot worked in the austenitic conditions. During hot working, small TiC precipitates form on the dislocations generated by the working. When cooled to ambient temperature, martensite forms; finally, the steel is tempered. Transmission electron microscopy of the A-21 reveals a high number density of small TiC particles uniformly distributed in the matrix. The strength of the

A-21 is less than the average value for modified 9Cr-1Mo below 600°C, but is greater above 600°C. In a Charpy impact test, the transition temperature of A-21 is similar to that of modified 9Cr-1Mo, but the upper-shelf energy is higher. Because of the fine TiC particles in the matrix, the creep-rupture properties of A-21 are superior to those of conventional Cr-Mo or reduced-activation Cr-W steels. Although the composition of the A-21 is not applicable for fusion because of the cobalt, the innovative production process may offer a route to an improved steel for fusion.

- 3.3 ON HYDROGEN AND HELIUM EMBRITTLEMENT IN ISOTOPIC TAILORING EXPERIMENTS** – D. S. Gelles, M. L. Hamilton, B. M. Oliver, and L. R. Greenwood (Pacific Northwest National Laboratory) 149
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- The tensile properties and room temperature electrical resistivity have been measured for pure copper and MAGT 0.2, GlidCop AL15 and GlidCop AL25 oxide dispersion strengthened copper alloys following irradiation in the spectrally tailored HFIR-MFE-200J and -400J irradiation capsules. The tensile measurements were performed at the irradiation temperature and at room temperature, at strain rates between $2 \times 10^{-5} \text{ s}^{-1}$ and 0.01 s^{-1} . Significant increases in the tensile strength were observed following irradiation at 200°C, accompanied by a reduction in uniform elongation. The irradiation at 400°C had only a slight effect on the tensile properties. A large increase in the electrical resistivity (attributable to solid solution transmutation products) was observed for both irradiation temperatures. The tensile strength showed a slight increase with increasing strain rate, in agreement with previous observations in copper. ****SUMMARIZE MAIN RESULTS—change in resistivity, presence of flow localization/ loss of strain hardening capacity in 200°C specimens; mention microstructural results obtained in Risø.**
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The requirements of this task are to complete the engineering designs of irradiation capsules in BOR-60. The specimen matrix will include sheet tensile specimens, Charpy impact specimens, TEM disks, and pressurized creep tubes. This experiment will not include DHCE samples. To better utilize the test volume and provide additional temperature options, it was decided to modify the experiment from a two-capsule to a three-capsule design. All capsules will be liquid-metal-

bonded for temperature uniformity. The top two capsules will be fitted with thermocouples for temperature measurement in early stages of the irradiation. Goal temperatures for the three capsules will be 450, 600, and 700-750°C, with an emphasis on 600°C. A key objective of the experiment will be to generate irradiation creep data for vanadium-base alloys, especially at the emphasized temperature of 600°C, where thermal creep may not be dominant. To ensure correct generation of irradiation creep data, knowledge of the temperature and minimal temperature fluctuations during irradiation are important.

- 11.2 HIGH-SENSITIVITY QUADRUPOLE MASS SPECTROMETER SYSTEM FOR THE DETERMINATION OF HYDROGEN IN IRRADIATED MATERIALS** – B. M. Oliver, F. A. Garner, L. R. Greenwood, and J. A. Abrefah (Pacific Northwest National Laboratory) 249

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- 11.3 SCHEDULE AND STATUS OF IRRADIATION EXPERIMENTS** – A. F. Rowcliffe (Oak Ridge National Laboratory) 250

The current status of reactor irradiation experiments is presented in tables summarizing the experimental objectives, conditions, and schedule.