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1.1 OXIDATION OF V-4Cr-4Ti AT LOW PRESSURES — B. A. Pint, R. DiStefano, and L. D. Chitwood (Oak Ridge National Laboratory) 3

Specimens of V-4Cr-4Ti have been exposed to low pressure oxygen and high purity helium and argon environments to determine oxidation kinetics. After exposure, mechanical properties were measured at 25 and 600°C. Oxidation at 600-700°C generally produced linear kinetics when the oxygenpressure was $\leq 10^{-5}$Pa. Higher oxygen partial pressures and lower temperatures can result in external film formation and parabolic kinetics. Internal oxidation resulting in oxygen concentrations >2000 wppm generally led to significant tensile embrittlement. Higher total oxygen levels could be tolerated (without significant embrittlement) in cases where an external oxide layer formed. However, when specimens which formed an external layer were given an additional vacuum anneal for 2000 h at 700°C, embrittlement occurred. This indicates that, rather than being protective, surface oxides on vanadium are a source of oxygen for further internal oxidation and thus embrittlement.

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1.3 LASER WELDING OF V-4Cr-4Ti ALLOY — Z. Xu, d. L. Smith, Y. Yan, and C. B. Reed (Argonne National Laboratory) 14

Laser welding offers potential advantages for welding vanadium alloys, including increased flexibility for field and large component welding with acceptable atmospheric control. A pulsed Nd:YAG laser with a fiber optic delivery system is used to conduct a systematic investigation of the weld parameters and environmental control requirements to obtain high intergrity laser welds of vanadium alloys. Ths current effort is focused on determination of laser weld parameters for welding ~4-mm-thick plate of V-4Cr-4Ti alloys and evaluation of various approaches for providing adequate environmental control. Laser weld specimens have been produced on two heats of V-4Cr-4Ti alloys and preliminary evaluations of weld integrity have been performed. Parameters for obtaining full penetration of welds have been defined and weld specimens have been prepared

for mechanical testing in a related study. Results from preliminary bend tests on weld specimens indicate good ductility.

1.4 MICROSTRUCTURAL CHARACTERIZATION AND IMPACT PROPERTIES OF V-4Cr-4Ti LASER WELDMENTS — Y. Yan, H. Tsai, A. D. Storey, D. L. Smith, and Z. Xu (Argonne National Laboratory) 17

Microstructure and Charpy-impact properties of laser weldments of the 500-kg #832665 heat V-4Cr-4Ti alloy were investigated. Optical metallography shows the grain sizes in the weld to be greater than those in the base materials. The grains are equiaxed near the middle of the weld and elongated (oriented in the direction of freeze progression) near the edge. The banded structure in the base metal seems to disappear in the weld zone, and density of secondary phase particles in the weld is much lower than that in the base metal. Impact testing was performed with specimens as-machined (by electric discharge machining with water as the flushing fluid), as well as after a degassing heat treatment at 400°C for 1 h. In the as-machined condition, the ductile-to-brittle-transition temperature (DBTT) for both the base metal and the laser weld is about –20°C. While the DBTT of the base metal was decreased significantly with the degassing operation, that of the weldment was relatively unaffected. Additional characterization and testing, including oxygen analysis is under way.

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Two candidate materials for insulating coatings in a lithium-cooled fusion reactor have been exposed to lithium in 100 h isothermal tests from 400 to 800°C to determine their maximum compatibility temperature. Bulk samples of AlN+5wt%Y_2O_3 showed significant mass loss at 600°C and higher temperatures. The amount of attack was reduced when AlN+0.4Y was tested. Characterization by auger spectroscopy of a AlN+-0.4Y specimen exposed at 600°C indicated the possibility of a lithium aluminate compound formation. Bulk specimens of CaO (99.9% purity) showed mass losses above 500°C indicating a possible dissolution problem that had not been observed in previous short-term screening tests. Doping of the lithium with oxygen (in the case of CaO) did not appear effective in reducing the attack at 600°C. However, doping with nitrogen (with AlN) did show a possible beneficial effect in conjunction with a Mo capsule. Based on these results, future testing also will consider alternative candidates such as Y_2O_3 and Er_2O_3.

1.7 DEVELOPMENT OF ELECTRICALLY INSULATING CaO COATINGS — K. Natesan, Z. Zeng, W. E. Ruther, M. Uz, and D. L. Smith (Argonne National Laboratory) 30

A systematic study has been initiated to develop electrically insulating CaO coating by in-situ formation in a liquid Li environment. Twelve capsule tests were conducted at several temperatures and with different concentrations of Ca in Li-Ca mixtures. Specimens included in the run were annealed V-4Cr-4Ti alloy without and with a grit-blasted surface, O-precharged in 99.999 vol.% Ar environment, polished specimens precharged in a 99.999 vol.% Ar, and 5000 vppm O_2-N_2 mixture. CaO coatings of 2-3 μm in thickness were obtained. Detailed microstructural and compositional analyses were performed on the exposed specimens. The coatings exhibited a resistance of ≈44 Ω/cm^2 on the surface of V-4Cr-4Ti alloys.

A systematic vapor transport study has been in progress to develop electrically insulating CaO coatings that are compatible with use in a liquid Li environment. Several additional experiments were conducted to study how the deposition of Ca on V-4Cr-4Ti substrate alloys is affected by variations in process temperature and time, and specimen location, surface preparation, and pretreatment. During this reporting period, several specimens were prepared with a coating of CaO by thermal/chemical deposition and the coatings were characterized before and after exposure to an Li environment.

1.8 UNIAXIAL CREEP BEHAVIOR OF V-4Cr-4Ti ALLOY — K. Natesan, W. K. Soppet, and D. L. Rink (Argonne National Laboratory) 39

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 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. The Larson-Miller approach is used to correlate several creep parameters such as time to rupture, time-to-inset of tertiary creep, and times for 1 and 2% strain accumulation with applied stress and temperature. Best-fit equations are presented for several creep parameters.

1.9 EVALUATION OF THERMAL CREEP OF V-4Cr-4Ti IN A LITHIUM ENVIRONMENT — M. L. Grossbeck (Oak Ridge National Laboratory) 48

Thermal creep in vanadium alloys can be measured by pressurized tubes. The tubes are pressurized with helium at a series of pressures to give the desired stresses in the tube wall. The diameter of the tubes is very precisely measured before and after high temperature exposure to determine creep deformation. A pair of high temperature furnaces and retorts have been fitted into an inert gas glove box system. Liquid lithium will be contained in the retorts which will contain the pressurized tubes of the vanadium alloy to be tested. The refractory metal retorts have been equipped with pressure sensors to determine to time of tube failure. This system will be used to determine time of failure and strain to failure as a function of stress.

1.10 DEFORMATION BEHAVIOR OF UNALLOYED VANADIUM – D. T. Hoelzer and A. F. Rowcliffe 56

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1.11 TENSILE PROPERTIES OF V-(Cr,Fe)-Ti ALLOYS AFTER IRRADIATION IN THE HFIR-11J EXPERIMENT — Y. Yan, H. Tsai, D. O. Pushis, and D. L. Smith (Argonne National Laboratory), K. Fukumoto, H. Matsui (IMR/Tohoku University) 62

Postirradiation tensile tests at room temperature and 300°C were performed on V-(Cr,Fe)-Ti alloy specimens that had been irradiated in the HFIR-11J experiment. The specimens were of the SSJ design. Irradiation temperature was ≈300°C and the attained neutron damage was ≈6 dpa. Results from these tensile tests show significant radiation hardening and ductility reduction in all of the materials. In comparison, hardening was substantially lower in the same materials after the 500°C 12J experiment. These results are consistent with previous findings that the demarcation for low-temperature radiation embrittlement in vanadium alloys is ≈400°C.

1.12 IMPACT AND TENSILE PROPERTIES OF VANADIUM ALLOY AFTER LONG-TERM EXPOSURE IN THE DIII-D EXPERIMENTAL TOKAMAK — H. Tsai, Y. Yan, D. O. Pushis, A. Storey, D. L. Smith (Argonne National Laboratory), and W. R. Johnson (General Atomics) 68

Tensile and Charpy specimens of Heat 832665 of V-4Cr-4Ti alloy were exposed in the DIII-D experimental tokamak to investigate the effect of environment on the impact and tensile properties of this alloy. In this last test of the four-test series, specimens were mounted next to the chamber wall behind a divertor baffle plate and received an exposure of ≈3 yr. The results of this test, consistent with the results of the previous three tests, indicate no significant degradation of the mechanical properties of the alloy.

1.13 EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE OF V-4Cr-4Ti MATERIALS — Y. Yan, H. Tsai, W. C. Kettman, and D. L. Smith (Argonne National Laboratory) 72

A banded structure containing Ti-rich particles has been observed in an extruded V-4Cr-4Ti bar from the ANL's 832665 heat and in a swaged rod from the GA's 832864 heat. Because these two materials may be used as feedstocks for the upcoming creep tubing fabrication, this task was undertaken to determine whether this secondary-phase structure could be removed by dissolution at elevated temperature. Small pieces of the two mateirals were heat treated at 1150°C for 3 h and then characterized. The preliminary results show that this heat treatment did not redissolve the banded structure in either the extruded 832665 bar or the swaged 832864 rod.

1.14 TOUGHNESS-TEMPERATURE CURVES IN OXIDE DISPERSION STRENGTHENED MA957 -- M.J. Alinger, G.R. Odette and G.E. Lucas (University of California, Santa Barbara) 77

Iron based alloys strengthened with a very high density and fine dispersion of ultra-fine scale yttria-titanium-oxygen particles for improved high temperature creep strength, coupled with chromium additions for corrosion resistance, offer great promise for elevated temperature applications in fusion reactors. These alloys are usually referred to as oxide dispersion strengthened (ODS) steels. They are typically produced through mechanical alloying by mixed powder attrition, followed by consolidation and hot deformation, which may include the production of near net shape components. Given their engineered microstructural architectures and low carbon content (a key factor in their success), we feel it is more appropriate to refer to this class of materials as nano-composited ferritic alloys (NFAs).

Mechanically alloyed 14Cr-1Ti-0.3Mo-0.025Y_2O_3 MA957 in the form of extrusions, were produced and extensively characterized for the breeder reactor program [1]. This material showed very promising creep and tensile properties. However, limited Charpy testing demonstrated that this MA957 product has highly anisotropic grain structures and manifests extremely brittle behavior, particularly in certain orientations. Brittle behavior was attributed to the presence of alumina stringers. The objective of the present work is to characterize the fracture toughness-temperature curves for MA957 in various orientations relative to the grain and inclusion structures. Small, pre-cracked, 1/3-sized Charpy specimens of MA957 tested in three point bending under quasi-static conditions showed low minimum toughness (<20MPa$\sqrt{m}$) and lower transition cleavage up to temperatures around 50-70°C in both the C-R and C-L orientations. At higher temperatures these orientations also manifested low resistance to stable crack growth, with maximum load toughness values ranging from about 50-100 MPa$\sqrt{m}$. However, the cleavage regime is shifted to much lower temperatures of around -70°C for the L-R orientation; and at high temperature maximum load toughness increased to about 125 to 145 MPa$\sqrt{m}$. At intermediate temperatures the fracture process was complex, typically involving out of plane cracking and combinations of stable extension and small cleavage pop-in events. The latter results are very encouraging and suggest that with proper development, NFA systems offer great promise to combine very high strength with good toughness and corrosion resistance.

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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. Recently families of polymers that yield inorganic materials subsequent to pyrolysis have been a subject of extensive international research [2-9]. One such polymer, allyl-hydridopolycarbosilane (aHPCS), has been shown to have good high temperature

mechanical properties as well as requiring relatively low temperature for pyrolysis [5]. In this study the mechanical properties of joints fabricated by aHPCS were measured. Preliminary results indicate that the strength value of the aHPCS joints are slightly lower than those of reaction-based methods.

2.2 TENSILE STRENGTH AND FRACTURE SURFACE CHARACTERIZATION OF HI-NICALON™ SiC FIBERS — G. E. Youngblood, Charles Lewinsohn, and R. H. Jones (Pacific Northwest National Laboratory), and A. Kohyama (Institute of Advanced Energy, Kyoto University) 96

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The change in concentrations of elements due to transmutations resulting from neutron irradiation in the first wall of the ARIES-IV conceptual fusion energy device were determined as a function of neutron dose. SiC burns out at a rate of about 0.5% per effective full power year. The largest impurity concentration is that of He, but several other elements burn in at rates of hundreds of appm/efpy.

2.4 THE KFIB EXPERIMENT — G. E. Youngblood, D J. Senor and R. H. Jones (Pacific Northwest National Laboratory), W. Kowbel (MER Corporation), and A. Kohyama (Kyoto University) 102

Several rod-shaped specimens with uniaxially packed fibers (Hi-Nicalon™, Hi-Nicalon™ Type S, Tyranno™ SA and Amoco K1100™ types) in a pre-ceramic polymer matrix were fabricated. By using appropriate analytic models, the bare fiber thermal conductivity (K_f) will be determined as a function of temperature up to 1000°C before and after irradiation for samples cut from these rods. Preliminary thermal conductivity data for unirradiated fibers (Hi-Nicalon™ and Tyranno™ SA-B SiC and K1100™ graphite) and for three types of unirradiated composites made from these fibers (2D-Nicalon S/SiC multilayer/CVI-SiC, 3D-Nicalon S/PIP-SiC, and 2D-8HS Tyrannohex™ HP) are presented.

2.5 THE TRANSVERSE THERMAL CONDUCTIVITY OF 2D-SIC$_F$/SIC COMPOSITES — G. E. Youngblood, David J. Senor, R. H. Jones (Pacific Northwest National Laboratory), and Samuel Graham (Sandia National Laboratories) 109

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A new type of SiC composite called Tyrannohex™ is potentially suitable as a fusion reactor structural material. Tyrannohex™ composite plates are made by hot-pressing layups of Tyranno™ SA precursor fibers into various 1D and 2D configurations. The fiber-bonded composite plates contain nearly 100% fiber volume, so take advantage of the outstanding high temperature strength and creep properties of the Tyranno™ SA fiber, a nearly stoichiometric SiC fiber. The hot-

pressed plates are dense, strong, rigid, tough, thermally conductive and have high temperature stability.

The microstructure and thermal conductivity of a SA-Tyrannohex™ material with a 2D-woven configuration was evaluated prior to irradiation testing. The microstructure contained some small, flat interlaminar pores and intrabundle needle-like pores, and the transverse thermal conductivity was 25 and 21 W/mK at ambient and 1000°C, respectively. These results suggest that careful control of the fiber-bonded interlayers and the fiber architecture are critical to achieve both high thermal conductivity and toughness in Tyrannohex™ type materials.

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The microstructure and the tensile and creep behavior of two oxide dispersion-strengthened (ODS) steels—F.E-12Cr-0.25Y_2O_3 (designated 12Y1) and Fe-12Cr-3W-0.4Ti-0.25Y_2O^3 (12YWT)— were investigatd. Optical microscopy, transmission electron microscopy, and atom probe field ion microscopy studies indicated that the 12YWT contained a high density of extremely fine Y-Ti-O clusters, compared to the much larger oxide particles in the 12Y1. The fine dispersion of particles in the 12YWT gave it superior tnesile and creep properties.

3.2 TENSILE RESULTS OF LOW-ACTIVATION MARTENSITIC STEEL IRRADIATED IN HFIR RB-11J AND 12J SPECTRALLY TAILORED CAPSULTS — K. Shiba (Japan Atomic Energy Research Inatitute), R. L. Klueh (Oak Ridge National Laboratory), Y. Miwa, N. Igawa (Japan Atomic Energy Research Institute), and J. P. Robertson (Oak Ridge National Laboratory) 131

Post-irradiation tensile test results for F82H IEA heat base metal (BM) and TIG weldments are reported. Tensile specimens were irradiated to about 5 dpa at 300 and 500°C. Irradiation caused significant hardening at 300°C. The TIG weld metal (WM) exhibited almost the same amount of irradiation hardening as the BM, but the TIG weld joint (WJ) showed less hardening than BM and WM. Increasing the strain rate caused a slight increase in yield strength, while a decreasing strain rate caused a sharper stress drop after yielding for BM specimens irradiated at 500°C. However, a decrease in strain rate caused a reduction in elongation.

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3.5 INVESTIGATION OF THE MICROSTRUCTURE OF FRACTURE SPECIMENS FROM FIVE DIFFERENT PLATES OF THE 500 KG PROGRAM HEAT OF V-4Cr-4Ti -- E.G. Donahue, G.R. Odette, G.E. Lucas (University of California, Santa Barbara) 155

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The unirradiated tensile properties of wrought GlidCop AL25 (ITER grade zero, IG0), solutionized and aged CuCrZr, and wrought and aged CuCrNb have been measured at temperatures up to 700°C at strain rates between $4x10^{-4}$ s^{-1} and 0.02 s^{-1}. GlidCop Al25 exhibited rapid softening with increasing test temperature ≥300°C, whereas the CuCrZr and CuCrNb precipitation hardened alloys decreased more slowly with increasing temperature. The difference in tensile behavior of the GlidCop and precipitation-hardened alloys is attributed to the small grain size in GlidCop, which allows grain boundary sliding (Coble creep) to become the dominant deformation mode at temperatures above ~400°C.

4.2 OVERAGING OF OUTOKUMPU CuCrCr — D. J. Edwards (Pacific Northwest National Laboratory) and B. N. Singh (Risø National Laboratory) 176

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Single- or poly-crystalline specimens of SiC, Si_3N_4, MgO, Al_2O_3 and $MgAl_2O_4$ were implanted with 0.4-1 MeV H^+ or 0.4-1 MeV He^+ ion beams at room temperature and 650°C (~0.1 and 0.4 T_M) up to fluences of ~$1x10^{22}/m^2$. This produced peak implanted gas and displacement damage levels as high as ~50 at. % and 21 displacements per atom (dpa). The specimens were subsequently examined optically, and in cross-section using transmission electron microscopy. Subsurface blistering occurred for specimens irradiated to H^+ or He^+ fluences greater than about $3x10^{21}/m^2$ (~15 at. % implanted gas concentration), and surface exfoliation occurred for fluences above ~$1x10^{22}/m^2$. Helium was more effective at inducing blistering and exfoliation than H on an atomic basis. The threshold blistering and

exfoliation fluences for both ions decreased with increasing temperature. Both H^+ and He^+ were found to be very effective in inducing matrix cavity formation, due to their low solubility in these ceramics. Cavity formation was observed to initiate at the periphery of dislocation loops in several cases. The bubble formation and blistering behavior of the ceramics was similar to that observed for metals irradiated at comparable homologous temperatures.

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8.1 **COMPOSITIONAL AND TEMPERATURE DEPENDENCE OF VOID SWELLING IN MODEL FE-CR BASE ALLOYS IRRADIATED IN THE EBR-II FAST REACTOR** — B. H. Sencer and F. A. Garner (Pacific Northwest National Laboratory) 203

A series of annealed and aged Fe-xCr, Fe-12Cr-yC and Fe-12Cr-0.1C-zMo model alloys were irradiated in EBR-II at eight temperatures between 400 and 650°C and dose levels ranging from 35 to 131 dpa. Swelling-induced density changes observed in the binary alloys generally peaked at mid-chromium levels, with the chromium and temperature dependence expressed primarily in the duration of the transient regime. The steady state swelling rate at the lower irradiation temperatures was much higher than previous estimates, reaching ~0.2%/dpa and possibly still climbing at higher neutron exposures.

The dependence of swelling on molybdenum and carbon was more complex, depending on whether the temperature was relatively low or high. At temperatures of 482°C and above the effect of carbon additions was very pronounced, with swelling of Fe-12Cr jumping dramatically from near zero at 0.002%C to 6-10% at 0.1%C. This indicates that the major determinant of the composition and temperature dependence probably lies in the duration of the nucleation-dominated transient regime of swelling and not primarily in the steady-state swelling rate as previously envisioned. This raises the possibility that significant swelling may occur earlier in fusion and spallation neutron spectra where high gas generation rates may assist void nucleation.

8.2 RADIATION HARDENING IN HCP TITANIUM ALLOYS — D. J. Edwards (Pacific Northwest Laboratory), B. N. Singh and P. Toft (Risø National Laboratory) 214

Two titanium alloys were irradiated at 50 and 350°C to ~0.3 dpa. Tensile properties were determined for both alloys and related to the observed microstructure before and after deformation. Radiation hardening occurred in both alloys when irradiated at 50°C due to the presence of fine-scale defects, whereas at 350°C only the Ti-6Al-4V alloy showed significant hardening. Radiation-induced precipitation occurred in this alloy, yielding a high strength, lower ductile condition compared to the unirradiated condition.

8.3 PASSIVE TEMPERATURE MONITORS FOR THE HFIR METS EXPERIMENT — S. D. Connery (Rennselaer Polytechnic Institute), L. L. Snead (Oak Ridge National Laboratory), and D. Steiner (Renssalaer Polytechnic Institute) 221

The purpose of this work is to provide a method of indicating the maximum temperature in the uninstrumental Mapping Elevated Temperature Swelling (METS) capsules. Small heats of selected alloys and pure metals ("melt blocks") were fabricated with a range of melting temperatures to be included in the METS capsules. Visual inspection and/or radiography of the melt blocks after irradiation will indicate the maximum temperature attained to within ~20°C. METS subcapsules expected to remain below 1000°C will also include SiC temperature monitors. Post-irradiation annealing and dimensional analysis of the SiC temperature monitors will provide an estimate of the temperature at the end of irradiation to within ~45°C.

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An extensive database of atomic displacement cascades in iron has been developed using the method of molecular dynamics (MD). More than 300 simulations have been completed at 100K with energies between 0.1 and 100 keV, with a more limited range of simulations at 600 and 900K. This encompasses nearly all energies relevant to fission and fusion reactor irradiation environments since a 100 keV MD cascade corresponds to the average event following a collision with a 5.1 MeV neutron and an iron atom. Extensive statistical analysis of the database has determined representative average values for several primary damage parameters: the total number of surviving point defects, the fraction of the surviving point defects conatined in clusters formed during cascade cooling, and a measure of the size distribution of the in-cascade point defect clusters. The energy dependence of the MD-based primary damage parameters has been used to obtain spectrum-averaged defect productiong cross sections for typical fission and fusion reactor neutron energy spectra. Total point defect survival and the total fraction of either interstitials or vacancies in clusters exhibit little dependence on neutron energy spectrum, while the fraction of point defects in large clusters exhibits a potentially-significant spectrum dependence.

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No contributions.

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11.1 HFIR-MFE-RB-14J SPECIMEN LOADING LISTING AND OPERATIONAL SUMMARY – A. L. Qualls (Oak Ridge National Laboratory 241

The specimens are housed in three cylindrical holders within the experiment. The holders are centered about the HFIR mid-plane in a large unshielded RB irradiation location (approximately 21 cm from the reactor centerline) during irradiation.

The three holders are the main components of three independently controlled temperature regions. The upper region is positioned from 21.1 cm to 12.9 cm above the reactor centerline and is controlled to irradiate the specimens at 500°C. The middle region irradiates the specimens at 800°C. It is twice as long as the other two zones and is centered on the reactor mid-plane extending from 8.8 cm above the reactor centerline to -9.2 cm below the reactor centerline. The lower region is controlled to irradiate the specimens at approximately 300°C and is positioned from –12.4 cm to –21.4 cm below the reactor centerline. A schematic of the experimental region of the capsule is shown in Figure 1.

11.2 SUMMARY OF THE OPERATION OF THE VARYING TEMPERATURE EXPERIMENT — A. L. Qualls (ORNL), T. Muroga (NIFS) 266

The Varying Temperature Experiment (HFIR-MFE-RB-13J) ended irradiation after eight cycles in a europium shielded Removable Beryllium position in the HFIR reactor. The irradiation was terminated two cycles short of a planned ten-cycle irradiation due to a break in one of the capsule's gas supply lines, which occurred while the capsule was being handled during an outage. The break made it impossible to maintain constant operating pressure and steady temperature control could not be guaranteed. It was considered prudent to terminate the irradiation after 8 cycles, rather than risk a temperature excursion with 80% of the irradiation complete.