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- 6.2.2 SWELLING BEHAVIOR OF AUSTENITIC STAINLESS STEELS IRRADIATED AT 400°C IN ORR MFE-7J CAPSULE BY SPECTRALLY TAILORED NEUTRONS (Japan Atomic Energy Research Establishment and Oak Ridge National Laboratory) 152
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6.3.2	SWELLING DEPENDENCE OF NEUTRON-IRRADIATED VANADIUM ALLOYS ON TEMPERATURE, NEUTRON FLUENCE, AND THERMOMECHANICAL TREATMENT (Argonne National Laboratory and Tohoku University)	172
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Swelling of vanadium alloys was determined after irradiation at 420 and 600°C to neutron fluences ranging from 0.3×10^{27} neutrons/m² (17 @a) to 1.9×10^{27} neutrons/m² (114 dpa). Binary and ternary vanadium alloys with Cr, Ti, Mo, W, Ni, Fe, Zr, and Si additions were irradiated in either the fully annealed, partially annealed, or 10% cold-worked condition. Upon irradiation at 600°C, the swelling of vanadium to which Cr had been added was greatly exacerbated. Whereas the swelling of vanadium to which Ti, Mo, W, and Ni (3-20%) had been added was not significantly affected. Swelling of V-Cr alloys upon irradiation at 600°C was substantially reduced (<0.1% per dpa) by the addition of Ti (1-15%). Upon irradiation at 420°C, the swelling of the vanadium alloys was <0.2% per dpa. Partial annealing or 10% cold-working had no significant effect on swelling of the alloys.

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6.4.1	MECHANICAL PROPERTY CHANGES AND MICROSTRUCTURES OF DISPERSION-STRENGTHENED COPPER ALLOYS AFTER NEUTRON IRRADIATION AT 411, 414, AND 529°C (University of Illinois and Pacific Northwest Laboratory)	179
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Dispersion strengthened copper alloys have shown promise for certain high heat flux applications in both near term and long term fusion devices. This study examines mechanical properties changes and microstructural evolution in several oxide dispersion strengthened alloys which were subjected to high levels of irradiation-induced displacement damage. Irradiations were carried out in FFTF to 34 and 50 dpa at 411-414°C and 32 dpa at 529°C. The alloys include several oxide dispersion-strengthened alloys based on the Cu-Al system, as well as ones based on the Cu-Cr and Cu-Hf systems. Of this group, certain of the Cu-Al alloys, those produced by an internal oxidation technique to contain alumina weight fractions of 0.15 to 0.25% outperformed the other alloys in all respects. These alloys, designated CuAl15, CuAl20, and CuAl25, were found to be resistant to void swelling up to 50 dpa at 414°C, and to retain their superior mechanical and physical properties after extended irradiation. The major factor which controls the stability during irradiation was found to be the dispersoid volume fraction and distribution. The other alloys examined were less resistant to radiation-induced properties changes for a variety of reasons. Some of these include dispersoid redistribution by ballistic resolution, effects of retained dissolved oxygen, and nonuniformity of dispersion distribution. The effect of laser welding was also examined. This joining technique was found to be unacceptable since it destroys the dispersoid distribution and thereby the resistance of the alloys to radiation-induced damage.

6.4.2	BRAZING OF COPPER-ALUMINA ALLOYS (Auburn University)	196
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An induction braze has been developed to join copper-alumina alloys. This brazing technique is proposed to replace current furnace brazing methods that have yielded poor results because of silver ingress along the fine grain boundaries of the copper-alumina alloys. The induction braze (because of the short braze time) severely restricts silver ingress along the grain boundaries of the alloy. Tensile tests of induction brazed lap joints fail in the base material rather than the braze indicating good braze properties.

6.4.3	A BRIEF REVIEW OF CAVITY SWELLING AND HARDENING IN IRRADIATED COPPER AND COPPER ALLOYS (Oak Ridge National Laboratory)	201
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The literature on radiation-induced swelling and hardening in copper and its alloys is reviewed. Void formation does not occur during irradiation of copper unless suitable impurity atoms such as oxygen or helium are present. Void formation occurs for neutron irradiation temperatures of 180 to 550°C, with peak swelling occurring at ~320°C for irradiation at a damage rate of 2×10^{-1} dpa/s. The post-transient swelling rate has been measured to be ~0.5%/dpa at temperatures near 400°C. Dispersion-strengthened copper has been found to be very resistant to void swelling due to the high sink density associated with the dispersion-stabilized dislocation structure. Irradiation of copper at temperatures below 400°C generally causes an increase in strength due to the formation of defect clusters which inhibit dislocation motion. The radiation hardening can be adequately described by Seeger's dispersed barrier model, with a barrier strength for small defect clusters of $a \approx 0.2$. The radiation hardening apparently saturates for fluences greater than $\sim 10^{24}$ n/m² (~0.1 dpa) during irradiation at room temperature due to a saturation of the defect cluster density. Grain boundaries can modify the hardening behavior by blocking the transmission of dislocation slip bands, leading to a radiation-modified Hall-Petch relation between yield strength and grain size. Radiation-enhanced recrystallization can lead to softening of cold-worked copper alloys at temperatures above 300°C.

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6.5.1	ELECTROCHEMICAL AND MICROSTRUCTURAL CHARACTERIZATION OF AN AUSTENITIC STAINLESS STEEL IRRADIATED BY HEAVY IONS AT 515°C (Oak Ridge National Laboratory and Japan Atomic Energy Research Institute)	219

The electrochemical and microstructural behavior of a solution-annealed, heavy-ion-irradiated, austenitic stainless steel, designated LS1A, have been investigated at 515°C after doses of 1, 10, and 30 displacements per atom (dpa). Changes in electrochemical properties due to radiation induced segregation in thin radiation-affected layers of the material were detected by the electrochemical potentiokinetic reactivation (EPR) technique using transmission electron microscopy (TEM) disk specimens. At all doses, the Flade potential and reactivation charge were greater than those measured for thermally aged control specimens. Flade potentials increased with increasing dpa while the reactivation charge did not increase beyond 10 dpa. Grain face etching, similar to that found on EPR-tested neutron-irradiated austenitic stainless steels, was observed on all specimens after testing. Grain boundaries were etched after doses of 10 and 30 dpa. The extent of grain face etching increased with increasing dose. Duplicate heavy-ion-irradiated specimens were also examined by high resolution analytical electron microscopy (AEM). The 1-dpa specimen showed only a high density of small faulted dislocations (~10 nm), and no grain boundary precipitation was observed nor was any grain boundary segregation detected. AEM confirmed chromium depletion at grain boundaries as measured by EPR for the 10- and 30-dpa specimens. Depletion widths of approximately 10 nm and grain boundary chromium compositions of less than 10% by weight were found. Molybdenum was also depleted near the boundary, whereas nickel, silicon, and iron were enriched for the 10- and 30-dpa irradiations. Precipitation of G and eta phases and larger dislocation loops (~60 nm) were observed in the 10- and 30-dpa specimens.

6.5.2	ENVIRONMENTAL EFFECTS ON AQUEOUS STRESS CORROSION OF CANDIDATE AUSTENITIC STEELS FOR ITER STRUCTURAL APPLICATIONS (Argonne National Laboratory)	228
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Susceptibility of Types 316 NG, 316, and 304 stainless steel to SCC was investigated at several temperatures between 50 and 150°C in slow-strain-rate-tests (SSRTs) in oxygenated water that simulates important parameters anticipated in first-wall/blanket systems. The water chemistry was based on a computer code that yielded the nominal concentrations of molecular radiolytic species produced in an aqueous environment under conditions likely to be found in the ITER. To be conservative, however, the SSRTs were performed in a less benign, more oxidizing reference environment. Predominantly ductile fracture was observed in crevice specimens of Type 316 stainless steel (SS) strained to failure in a reference ITER water chemistry. The failure behavior of Type 304 SS crevice specimens heat-treated to yield sensitization values of 2, 3, or 20 C/cm² by electrochemical potentiokinetic reactivation (EPR) demonstrated that degree of sensitization had a dramatic effect on intergranular SCC (IGSCC) susceptibility. Type 304 SS specimens sensitized to the highest value exhibited shorter failure times, lower maximum stresses, and lower reduction in area values than did less-sensitized Type 304 SS or Type 316 NG SS specimens. Scanning electron microscopy showed minimal evidence of SCC in the Type 316 NG and solution-annealed Type 316 SS but clear evidence of IGSCC in moderately sensitized (EPR = 20 C/cm²) Type 304 SS specimens in these environments. Ranking for resistance to SCC in simulated ITER Water by electron microscopy and SSRT parameters (e.g., total elongation, ultimate strength, and reduction in area) is: 304 SS (EPR = 20 < 2 C/cm²) < 316 NG SS.

6.5.3	SEGREGATION IN THERMALLY AGED TYPE 304L ALLOYS FOR THE ICG-IASCC ROUND ROBIN (Oak Ridge National Laboratory)	236
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Grain boundary segregation in thermally aged type 304 stainless steels has been investigated by X-ray microanalysis. In one commercial alloy sensitized at low temperature, narrow (<10 nm) zones depleted of chromium, silicon, and molybdenum have been observed along with enrichment of phosphorus and nickel. For high purity alloys doped with either sulfur or phosphorus, no significant segregation of either impurity was detected. In a second commercial alloy which exhibited no grain boundary precipitation, segregation of phosphorus, chromium, and molybdenum to boundaries was observed along with a corresponding depletion of iron. The occurrence of phosphorus segregation in the two commercial alloys and absence in the high-purity, phosphorus-doped alloys seems to indicate some synergism of phosphorus Segregation with either chromium or carbon segregation or with the precipitation of M₂₃C₆ at the grain boundaries.

6.5.4	MEASUREMENT OF RADIATION-INDUCED SEGREGATION IN NEUTRON-IRRADIATED AUSTENITIC STAINLESS STEELS (Oak Ridge National Laboratory and Japan Atomic Energy Research Institute)	239
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X-ray microanalysis of RIS to grain boundaries and large dislocation loops in neutron-irradiated USPCA has indicated enrichment of silicon and nickel and depletion of chromium and iron. However, there are numerous artifacts which can lead to erroneous results and must be recognized and avoided. Some of these effects include surface films, preferential etching, overlapping RIS profiles from different defects, as well as the more obvious hole count and induced radioactivity or neutron-irradiated material. For irradiated EP838, silicon, nickel, and iron are enriched at boundaries, whereas manganese replaces chromium as the strongly depleted element. Preliminary measurements of RIS profiles at grain boundaries by parallel electron energy loss spectroscopy (PEELS) are discussed. The relative advantages and disadvantages of EDS and PEELS for such measurements are considered.

- 6.5.5 RADIATION-INDUCED GRAIN BOUNDARY SEGREGATION AND SENSITIZATION OF A NEUTRON-IRRADIATED AUSTENITIC STAINLESS STEEL (Oak Ridge National Laboratory and Japan Atomic Energy Research Institute) 244

Analytical electron microscopy and electrochemical potentiokinetic reactivation (EPR) testing were applied to the radiation-induced segregation (RIS) and sensitization of a titanium-modified austenitic stainless steel irradiated to 9 dpa at 420°C in the Materials open Test Assembly (MOTA) of the Fast Flux Test Facility (FFTF). The EPR testing of both solution annealed (SA) and 25% cold-worked (CW) materials indicated a significant increase in the reactivation charge (P_2). Both optical and scanning electron microscopy of the specimen surface after EPR testing indicated preferential attack at grain boundaries, indicative of sensitization. In addition, localized attack of the matrix was observed. Though precipitates were occasionally present on grain boundaries, they were not chromium-rich $M_{23}C_6$, but nickel- and silicon-enriched G phase. Faulted dislocations, fine γ' precipitates, and isolated cavities were observed in the matrix. X-ray microanalysis indicated significant RIS at high-angle boundaries in both materials. Depletion of chromium to apparent levels of 10 at. % was observed in the irradiated SA material. The boundaries were enriched in nickel, silicon, and titanium (up to 28, 6, and 1 at. %, respectively) and depleted of iron and molybdenum (as low as 54 and 0.7 at. %, respectively). The width of the Segregation zone was very narrow (<6 nm). Similar grain boundary RIS was observed in the Cold-worked material. There was also evidence for boundary migration in the cold-worked material (boundary faceting and asymmetric composition profiles). Voids and faulted dislocation loops in the SA material also exhibited similar RIS.

- 6.5.6 DEVELOPMENT OF CERAMIC COATINGS FOR LIQUID METAL BLANKET APPLICATIONS (National Chung Hsing University and Argonne National Laboratory) 252

Based on a preliminary survey of more than 15 oxides and nitrides, four materials (CaO , MgO , Y_2O_3 , and BN) were identified as candidates for insulator coating development. Additionally, Cr_2O_3 , V_2O_5 , and $(V,Ti)_xN$ were included for study because of their chemical stability in liquid lithium and their potential as corrosion inhibitors. These ceramic compounds were fabricated by a variety of techniques and exposed to flowing lithium at 400°C to assess chemical compatibility. Preparation technologies included hot-press-sintering of MgO , CaO , and Y_2O_3 ; diffusion coating of Cr_2O_3 ; oxidation of vanadium; and reactive sputtering of BN . Among the three hot-pressed-sintered materials, only Y_2O_3 displayed acceptable corrosion resistance when exposed to high purity lithium. A ferritic substrate alloy (HT-9, coated with a 1- μ m-thick BN layer) was immersed in flowing lithium at 400°C for 1 h, after which its physical stability and chemical compatibility were determined to be unacceptable. Chromized V-20Ti and oxidized V-20Ti immersed in flowing lithium at 410°C for 100 h showed greater potential for further development than did the BN insulator coating. Preliminary work has begun in the development of in-situ-formed insulator coatings. The first step has been to examine the characteristics of $(V,Ti)_xN$ reaction product layers formed after exposure of V-Ti specimens to flowing lithium. Work is also in progress to test a self-regenerating $CaO \cdot V_2O_5$ -type insulator coating on a vanadium substrate.

7. SOLID BREEDING MATERIALS 261

- 7.1 LITHIUM MASS TRANSPORT IN CERAMIC BREEDER MATERIALS (Argonne National Laboratory) 263

The transport of lithium by vaporization of $LiOH(g)$ from lithium ceramics, particularly $Li_2O(s)$, poses a constraint on the maximum operating temperature of the blanket. Experimental measurements have shown that, depending on temperature, moisture pressure, and proximity of structural steels, the lithium transport process is complex. For conditions wherein the $Li_2O(s)$ is "free standing," the lithium vaporization is controlled by the Li_2O/H_2O system thermodynamics that are already well established. Simply stated lithium transport as $LiOH(g)$, increases with increasing temperature and higher partial pressures of moisture. In the proximity of stainless steel, there is an added chemical potential driving force due to formation of Li_2CrO_2 , $LiFeO_2$ and $LiNiO_2$. The transport of $LiOH(g)$ to the stainless steel is driven by the concentration gradient of $LiOH(g)$ from that at the $Li_2O(s)$ surface and that at the steel surface. This gas-solid reaction may become important for blanket designs where the structural steel is very close to the $Li_2O(s)$ ceramic.

- 7.2 DESORPTION CHARACTERISTICS OF THE $LiAlO_2-H_2O(g)$ SYSTEM (Argonne National Laboratory) 268

The energetics and kinetics of the evolution of $H_2O(g)$ and $H_2(g)$ from $LiAlO_2$ are being studied by the temperature programmed desorption technique. The concentrations of H_2 , H_2O , N_2 , and O_2 in a helium stream during a temperature ramp are measured simultaneously with a mass spectrometer. The amount of H_2 adsorption/desorption is small compared to the amount of H_2O adsorption/desorption. After prolonged treatment with helium containing 990 ppm H_2 at 400°C, H_2O evolution into the He- H_2 stream was observed during 473 to 1023 K (200 to 750°C) ramps at rates of 2 or 5.6 K/min. The different peak shapes reflecting this process were deconvoluted to show that they are composites of only 2 or 3 reproducible processes. The activation energies and pre-exponential terms were evaluated. The different behavior originates in the differences among different surface sites for adsorption. The interpretation of higher temperature peaks [above 873 K (650°C)] must still consider the possibility of contributions from interactions with the steel walls.

7.3 TRITIUM RELEASE FROM LITHIUM CERAMICS (Argonne National Laboratory, CEA/CEN, ENEA/ICRE, and Karlsruhe) 274

Recent in-pile tritium extraction experiments from lithium ceramics have investigated the effects of temperature, material microstructure, purge gas composition, and tritium generation rate on the kinetics of tritium release. The relationship between the tritium release characteristics and sample microstructure indicates desorption is the dominant mechanism controlling tritium release for the conditions investigated. Literature results indicate that this desorption step may be second order and the activation energy of desorption may vary significantly with surface coverage. The changes in inventory with variations in the key parameters mentioned above were investigated to determine the applicability of these reports to in-pile tritium release. The results suggest tritium release is first order, and that the desorption activation energy is dependent on the surface coverage.

7.4 IN-SITU TRITIUM RECOVERY FROM Li₂O IRRADIATED IN FAST NEUTRON FLUX - BEATRIX-II INITIAL RESULTS (Japan Atomic Energy Research Institute, Pacific Northwest Laboratory, and Chalk River Laboratories) 279

The BEATRIX-II experiment in FFTF is an in-situ tritium recovery experiment to evaluate the tritium release characteristics of Li₂O and its stability under fast neutron irradiation to extended burnups. This experiment includes two specimens: a thin annular ring specimen capable of temperature transients and a larger temperature gradient specimen. During the first 85 days of the operating cycle of the reactor, the tritium recovery rate of a temperature transient capsule was examined as a function of temperature, gas flow rate, gas composition and burnup. Temperature changes in the range from 500 to 650°C resulted in decreasing tritium inventory with increasing temperature. Lower gas flow rates resulted in slightly lower tritium release rates while gas composition changes affected the tritium release rate significantly, more than either flow rate or temperature changes. Three different sweep gases were used: He-0.1% H₂, He-0.01% H₂, and pure He. Decreasing the amount of hydrogen in the sweep gas decreased the steady-state release rate by as much as a factor of two.

8. CERAMICS 289

8.1 IEA WORKSHOP ON IN-SITU MEASUREMENT OF ELECTRICAL PROPERTIES OF IRRADIATED CERAMICS (Los Alamos National Laboratory) 291

This International Energy Agency workshop was held in Los Alamos, NM June 27-29, 1990. In attendance were 14 participants representing fusion ceramics programs in Spain, the U.K., Japan and the U.S. The workshop was divided into four sessions: Background, Current In-Situ Work, Plans and Goals for Future work, and Recommendations. Informal discussion sessions on various subjects were also included in the workshop. Studies of in-situ electrical properties have accelerated since the last gathering of this group, with recent findings demonstrating that dielectric breakdown as well as enhanced conductivity can be induced by concurrent irradiation. Based on present knowledge, recommendations were made to designers of NET and ITER on materials selection and anticipated performance, as well as on design-related considerations.

8.2 IN-SITU MEASUREMENT OF RADIATION INDUCED CONDUCTIVITY IN CERAMICS (Los Alamos National Laboratory) 295

This report describes our experimental plan and schedule for measuring the radiation induced conductivity (RIC) in ceramics while being irradiated with 3 MeV protons. In the initial experiments, dielectric constant and loss will be measured in pure and in Ti-doped sapphire. The measurements will be made at room temperature, at frequencies between 100 Hz and 10 MHz, with and without an applied DC bias, and as a function of radiation flux and fluence. The Ion Beam Materials Laboratory (IBML) ion source at Los Alamos will be used with beam currents up to 1 micro-ampere. We expect initial results to be available for the next semi-annual Progress Report.

8.3 MILLIMETER-WAVE TESTING OF ISOTOPICALLY ENRICHED ALUMINA (Los Alamos National Laboratory and Oak Ridge National Laboratory) 297

In a special fusion-neutron irradiation simulation experiment, ¹⁷O has been substituted for ¹⁸O in 99.5% alumina to be neutron-irradiated in the High Flux Isotope Reactor (HFIR) at Oak Ridge for raising the potentially deleterious helium gas yield to levels closer to those expected from the neutrons of deuterium-tritium (D-T) fusion reactions. Earlier, we reported values of complex dielectric constant k* measured at Los Alamos at millimeter-wave (MMW) frequencies for conventional (unenriched) 99.5% alumina test specimens. These specimens were made under the same conditions applying to the present report of k* measured for ¹⁷O-enriched specimens based on the same alumina system. The present alumina has lower dielectric losses than that reported in the earlier work. Furthermore, we corroborate the earlier finding that k* is a sensitive nondestructive measure of the quality of alumina being considered for rf-window use -- and the overall quality of high-purity ceramics in general.

8.4. WORKSHOP ON CERAMIC MATRIX COMPOSITE MATERIALS FOR STRWTURAL APPLICATIONS IN FUSION REACTORS (Pacific Northwest Laboratory and university of California-SB) 302

The workshop to assess the potential application of ceramic matrix composites (CMCs) for structural applications in fusion reactors was held on May 21-22, 1990, at University of California, Santa Barbara. Participants included individuals familiar with materials and design requirements in fusion reactors, ceramic composite processing and properties and radiation effects.

Clear advantages for the use of CMCs (i.e., SiC/SiC) are high-temperature operation, which would allow a high-efficiency Rankine cycle, and low activation. Limitations to their use are material costs, fabrication complexity and costs, lack of familiarity with these materials in design, and the lack of data on radiation stability at relevant temperatures and fluences. Fusion-relevant feasibility issues were identified.

8.5 DEVELOPMENT OF THIN-SECTION PUSH-OUT TECHNIQUE FOR USE IN MEASURING RADIATION-INDUCED MODIFICATION OF COMPOSITE INTERFACES (Rensselaer Polytechnic Institute and Oak Ridge National Laboratory) 306

A technique for measuring the interfacial properties for extremely thin composite sections is presented. The data shown is for a single composite section of 22 μm thickness. By employing a Nanoindenter microhardness tester, composite fibers were individually loaded and the debond and frictional Sliding Strength measured. It is shown that such a technique can discriminate between the debond and frictional components of the interfacial band. The technique presented is a substantial improvement over previous techniques in both thickness of composite section, fiber loading accuracy, and percentage of fiber failures. Though the data presented is only for a single section, the results are typical of other sections tested. A statistical analysis of the data suggests that a Weibull treatment is more appropriate to interfacial data than the commonly used normal distribution.

8.6 MEASUREMENT OF DIELECTRIC PROPERTIES IN ALUMINA UNDER IONIZING AND DISPLACIVE IRRADIATION CONDITIONS (Oak Ridge National Laboratory) 317

Several experiments have been completed in which the dielectric properties of alumina have been measured in the presence of ionizing and displacive irradiation. Spent fuel elements from the High Flux Isotope Reactor (HFIR) at the Oak Ridge National Laboratory were used to provide an intense source of ionizing irradiation for some of the measurements. The TRIGA reactor at the University of Illinois was used to provide an irradiation field that produced both ionization and atomic displacements. The results of these in situ measurements indicate that the dielectric properties of alumina are more severely degraded by displacive irradiation than was indicated by earlier, post-irradiation measurements.

8.7 DISLOCATION LOOP FORMATION IN ION-IRRADIATED POLYCRYSTALLINE SPINEL AND ALUMINA (Oak Ridge National Laboratory) 323

The microstructure of magnesium aluminate spinel (MgAl₂O₄) and alumina (Al₂O₃) has been examined with transmission electron microscopy following ion irradiation to damage levels of 0.1 to 5 keV/atom (1 to 50 dpa) at room temperature and 650°C. The ion irradiation produced interstitial dislocation loops of types a/4<110>{110} and a/4<110>{111} in spinel along with a very low density of a/6<111>{111} loops. Dislocation loops of types a/30001 and a/3<1100>{1100} were tentatively identified in alumina. The loop size increased and the density decreased gradually with increasing fluence in spinel irradiated at 650°C, with the net result that the concentration of interstitials contained in the loops remained nearly constant at ~0.1 at. %. Defect-free regions were observed adjacent to grain boundaries and the irradiated surface in spinel irradiated at 650°C. The denuded zone width was very small for spinel irradiated at 25°C and Al₂O₃ irradiated at 650°C. For a given irradiation temperature, the loops in spinel were larger and of much lower density than the loops in Al₂O₃.

8.8 TECHNIQUE FOR PREPARING CROSS-SECTION TRANSMISSION ELECTRON MICROSCOPE SPECIMENS FROM ION-IRRADIATED CERAMICS (Oak Ridge National Laboratory) 334

The general techniques necessary to produce high-quality ceramic specimen for transmission electron microscope observation are outlined. A particularly important point is that the width of the glued region between faces of the ceramic specimen must be <0.2 μm to prevent loss of the near-surface region during ion milling. A recently developed vise for gluing ceramic cross-section specimens is described, and some examples of the effect of glue thickness on specimen quality are shown.