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SEMI-ANNUAL PROGRESS REPORT
IRRADIATION EFFECTS ON REACTOR
STRUCTURAL MATERIALS
FEBRUARY, 1974 TO JULY, 1974

MASTER

HANFORD ENGINEERING DEVELOPMENT LABORATORY
Operated by Westinghouse Hanford Company
A Subsidiary of Westinghouse Electric Corporation

P.O. Box 1970 Richland, WA 99352

Prepared for the U.S. Atomic Energy Commission
Division of Reactor Research and Development
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Compiled by T. T. Claudson

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SUMMARY

AEROJECT NUCLEAR COMPANY

Results of strain controlled fatigue tests at 482°C (900°F) of irradiated (0.5 to 1.4×10^{21} n/cm², $E > 0.1$ MeV) and unirradiated 308 SS weld metal with controlled residual elements (CRE) are compared to the base metal. Also, the effects of irradiation to fluences of 0.3 to 1.0×10^{22} n/cm² ($E > 0.1$ MeV) are shown for the weld and base metal at 593°C (1100°F) with and without tensile hold periods. The unirradiated weld metal is inferior to the base metal at high strain ranges, but approaches the base metal at lower strain ranges. Irradiation further reduces the cyclic life in the high ranges, but the irradiated material becomes equivalent or slightly superior in the low strain ranges. As tensile hold periods are added to each cycle, the weld metal exhibits less reduction in cyclic life and becomes superior to the base metal in both the unirradiated and irradiated state as hold periods are increased. Irradiation causes a significant reduction in life with tensile hold periods.

The effects of cold work on the fatigue properties of irradiated 304 and 316 SS are being investigated. Tests conducted at 427°C (800°F) on material irradiated in the 10^{20} n/cm² range at 380 to 450°C showed no significant degradation due to CW-irradiation interaction. Tests are planned for higher fluence, higher temperature tests with hold times.

ATOMICS INTERNATIONAL

Swelling of 20% CW 316 stainless steel was determined as a function of damage up to 24 dpa for samples proton irradiated at 500°C and 600°C. The swelling varied linearly with damage and there was no reduction in the swelling rate at the highest damage levels.

Negligible void formation was observed in samples of 405 SS (ferritic) irradiated in EBR-II X100 at 500°C and 600°C to a fluence of 4×10^{22} ($E > 0.1$ MeV).

GENERAL ELECTRIC COMPANY--BREEDER REACTOR OPERATION

Irradiation-induced swelling in 304 and 316 SS was monitored by measuring diameter increases of unstressed tubular specimens after fluences of ~ 3.2 and $\sim 7.8 \times 10^{22}$ n/cm² $E > 0.1$ MeV in EBR-II. The results show that Type 304 SS exhibited more swelling than Type 316 SS for both solution-annealed and cold-worked conditions. The peak swelling temperature for 316 SS was $\sim 600^\circ\text{C}$, which was higher than that for 304 SS. The maximum swelling measured for 20% CW 316 SS was 1.72% or less than half that observed for annealed 316 SS.

Pre-exposure to flowing sodium was demonstrated to greatly increase the swelling in three austenitic stainless steels irradiated in EBR-II. Specimens of 316, 321 and 347 SS pre-exposed to 704°C for 15,000 hours experienced density decreases from 4% to 16% after $\sim 500^\circ\text{C}$ exposure to neutron irradiation for a fluence of $\sim 5 \times 10^{22}$ n/cm², $E > 0.1$ MeV. Control specimens not previously exposed to sodium exhibited density decreases of less than 2%.

NAVAL RESEARCH LABORATORY

I. ADVANCED REACTOR STRUCTURAL MATERIALS

A cooperative AEC-CE-NRL research program is exploring trends in increased 550°F (288°C) radiation embrittlement resistance with reduced copper content for commercially produced ASTM A533-B plates, welds, and weld heat affected zones. Objectives are: 1) to develop information assisting the formulation of specifications for improved steels for nuclear service; and 2) to delineate associated embrittlement resistance trends for vessel design and operation.

Materials selected for study depict three levels of copper content control as follows: Series 1 - $\geq 0.15\%$ copper, typical of nonimproved commercial steel production (pre-1971); Series 2 - 0.10% copper max., representing improved steel production; and Series 3 - 0.06% copper max., considered the practical lower limit for copper content control. Radiation assessments of the Series 1 (normal copper content) and Series 2 (low copper content) materials have been made and are summarized; investigations of the Series 3 materials are still in progress.

Observations indicate a clear benefit of low copper content to 550°F (288°C) radiation embrittlement resistance. Charpy-V 30 ft-lb transition temperature increases for low copper content plates and welds did not exceed 100°F (56°C) for fluences to 5×10^{19} n/cm² >1 MeV. New (proposed) ASTM and AWS specifications on allowable copper (and phosphorus) content of reactor vessel materials are shown justified.

Strength and Charpy-V notch ductility changes with elevated temperature, high fluence irradiation have been explored for several low alloy structural steels, including A302-B, A533-B, A543-1, 9Ni-4Co-.20C, 12-6PH, and 12Ni-5Cr-3Mo compositions. The irradiations were performed in the Advanced Test Reactor at 550°F (288°C); neutron fluences ranged from 1 to 7×10^{20} n/cm² >1 MeV.

Yield strength vs temperature relationships were compared for pre- vs post-irradiation conditions. Several important data trends were evident. In particular, the data patterns suggest that accurate projections of yield strength behavior for temperatures in the range 75 to 550°F (24 to 288°C) should be possible from only limited post-irradiation data.

The Ratio Analysis Diagram was applied to assess and compare post-irradiation fracture resistance in the upper shelf condition. An A533-B plate and weld deposit specially tailored for improved radiation resistance (low copper and low phosphorus contents) and a 12-6PH experimental alloy demonstrated particularly high fracture resistance at upper shelf temperatures after $\sim 2.5 \times 10^{20}$.

The toughness of 316 SS forging and weld metal in both irradiated and unirradiated conditions was compared using the J-Integral technique. Notched, three-point bend specimens having a/W ratios between 0.3 and 0.5 were located statically and J-Integral values were computed using an approximate expression. The R-curve technique was employed to define the critical J_{IC} value at crack initiation for tests conducted at 500°F (260°C). The J values more than doubled as a function of the slow stable (rising load) crack extension that occurred between the point of crack initiation, corresponding to J_{IC} , and the point of maximum load. A fluence of 5.3×10^{19} n/cm² >1 MeV was found to degrade an initially poor weld metal to the point where linear elastic K_{IC} values should be measurable in sections greater than 3 in.

(76 mm) in thickness. The toughness of the forging was significantly above that of the weld. Irradiation also reduced the toughness of the forging, but this material still retained an irradiated toughness that was above that of the unirradiated weld metal.

The microstructure and swelling of annealed 304 SS from EBR-II control rod thimble 5D3 were examined using TEM and immersion density methods. The material received neutron fluences between 0.9 and 16×10^{22} n/cm² >0.1 MeV at irradiation temperatures from 371 to 482°C (700 to 900°F). Unstrained portions of previously tested fatigue specimens were employed in the examination. Radiation-induced voids were observed in all specimens. The void number density and mean void diameter were found to be strongly dependent on irradiation temperature and neutron fluence. Comparisons of microstructures taken from specimens tested at 427 and 593°C indicate that thermal recovery of dislocations and loops has occurred at the higher test temperature. The maximum swelling of 10.4% determined by both microscopy and immersion density techniques occurred 3 in. above the core midplane. This position corresponds to a fluence of 1.4×10^{23} n/cm² and an irradiation temperature of 415°C (799°F). Comparison of the swelling determined from the immersion density measurements with that computed from an empirical correlation for 304 SS indicated that the empirical expression overestimated the maximum swelling by approximately 50%.

II. RADIATION DAMAGE MECHANISMS

The effect of ion dose on the void and dislocation structures of nickel-ion-bombarded nickel was studied. The specimens examined were irradiated at a temperature of 575°C and a damage rate of 7×10^2 displacements per atom per second to damage levels of 4, 13, 40, and 130 dpa. With increasing damage level, the void density decreased and the mean void diameter increased, while the dislocation density was relatively constant up to 40 dpa, but decreased by approximately a factor of 3 at 130 dpa. Analysis of the results by void growth theory indicated that to explain the observed dependence of the swelling on damage levels, the effective bias for interstitial capture had to be higher for the 4 dpa dislocation structure than the bias for the structures at 13 dpa and above. Implications of this result are discussed in the light of recent theoretical models.

As to the studies on helium mobilities in metals, work reported during this period is an extension of the helium mobility studies described in the last report. Additional experimental data on bubble formation in 316 SS and aluminum are reported. The bubble growth data at different temperatures are analyzed by both a bubble diffusion model and an Ostwald ripening model. The data are found to fit the bubble diffusion model best.

III. ARMY REACTOR STUDIES

Tensile specimens from a third series of MH-1A reactor vessel surveillance capsules have been evaluated. Results for specimens exposed at the vessel wall and at an above-core (thermal control) location indicate no appreciable strengthening of the vessel forging by irradiation. Some radiation strengthening was observed for specimens from accelerated exposure locations, but 75 and 500°F (24 and 260°C) yield strength values remained below 100 ksi. The difference between the yield and tensile strength even under these severe irradiation conditions is adequate.

OAKRIDGE NATIONAL LABORATORY

The effects of titanium additions up to 0.6 wt % on the irradiation-induced swelling and changes in creep-rupture properties have been investigated. Samples were irradiated in the EBR-II at temperatures in the range 450 to 700°C to a maximum neutron fluence of 7.8×10^{26} n/m² (>0.1 MeV). In annealed material, the irradiation-induced swelling exhibited a minimum in the range 0.2-0.4 wt % Ti. The minimum in swelling was directly attributable to a minimum in the concentration of voids. Samples irradiated in the 20% CW condition exhibited slight densification at 3.0×10^{26} n/m² (>0.1 MeV) at both 500 and 600°C. A small density decrease (0.23%) occurred during irradiation to 6.6×10^{26} n/m² (>0.1 MeV). Post-irradiation creep-rupture ductility was maximum for alloys containing 0.23 and 0.33 wt % Ti. The observed swelling behavior in the annealed material is thought to be associated with changing amounts of titanium and carbon in solution in the austenite as the total titanium concentration is increased. The improved ductility is attributable to a decreased tendency for grain-boundary crack formation and appears to be associated with removal of sulfur and possible other impurities from solution in the austenite.

Initial results on the effects of molybdenum concentration on swelling of 316 SS are presented. At 500 and 600°C, the density decrease increased monotonically with molybdenum for a fluence of 5.6×10^{26} n/m² (>0.1 MeV). Molybdenum also appears to influence the temperature-dependence of the swelling.

Density measurements and available creep-rupture results on 316 SS samples having variations in pre-irradiation microstructure are reported. Some microstructures have been found that have swelling resistance approximately equal to 20% CW 316 SS and retain creep-rupture elongation of ~5% after irradiation to 6.6×10^{26} n/m² (>0.1 MeV).

The high helium production rates expected during structural irradiation in controlled thermonuclear reactors can be approximated in nickel-containing alloys by irradiation in thermal spectrum fission reactors. Examination of 316 SS samples irradiated in HFIR to fluences approaching 10^{27} n/m² has yielded results that are different in important respects from irradiations to equivalent fluences in fast reactors. Optical metallography of irradiated material showed extensive precipitation in both annealed and CW samples. The amount and morphology of the precipitate was dependent on the irradiation temperature and was much more extensive than would have occurred in unirradiated thermal aging for equivalent times. The tensile test fracture mode was found to be intergranular for a sample irradiated in the annealed condition at 685°C and tested at 650°C. For irradiation at 380 and tests at 350°C, however, the fracture mode was predominantly transgranular.

PACIFIC NORTHWEST LABORATORIES

The void-induced swelling has been studied in molybdenum which has been bombarded with high energy tantalum ions at 900 to 1000°C. The dose dependence of the void parameters has been evaluated and compared with results from similar studies by Evans on nitrogen-bombarded molybdenum.

UNIVERSITY OF CINCINNATI

The fractional length change of molybdenum specimens irradiated to a fast neutron fluence of about $1 \times 10^{22} \text{ n/cm}^{-2}$ ($E_n > 1 \text{ MeV}$) at 430°C is about 0.32%. Annealing studies show that the major recovery peaks occur at about 750 and 1350°C , with the latter stage attributed to the removal of voids.

A program to determine the temperature and dose dependence of ion-induced swelling in molybdenum and several dilute alloys of molybdenum has been initiated. The irradiations were carried out on the ANL Dynamatron Facility using vanadium ions.

Neutron-induced swelling results in W and W-25Re show reasonable agreement when bulk density measurements are compared with dimensional measurements performed on the same samples.

Void sizes and void densities in niobium and molybdenum specimens were irradiated simultaneously at six temperatures in the EBR-II facility. The void size and density remained constant for niobium in the temperature regime of 0.26 to $0.36 T_m$, whereas for molybdenum, these parameters remain constant in the temperature regime of 0.24 to $0.34 T_m$. The void density decreases and the size increases for irradiation temperatures greater than $0.36 T_m$ for niobium and $0.34 T_m$ for molybdenum.

HANFORD ENGINEERING DEVELOPMENT LABORATORY

Sensitivity Analysis of History Effects Experiment H-3

The history effects experiment consists of irradiating a series of small stress relaxation and primary loaded beam specimens constructed of previously irradiated material. The deformation of these specimens as measured during interim examinations will be studied in order to determine the effects of prior irradiation history on irradiation creep. In the present work, an analysis was made to establish that the specimen designs considered will be sensitive enough to distinguish prior irradiation-induced changes in creep behavior.

Viscoelastic Analysis of Stress Relaxation in a Slit Tube

The time dependence of stress-relaxation in a slit tube is calculated on the basis of isotropic viscoelastic theory. The results of the calculation show that it is feasible to use slit tube stress-relaxation data to measure in-reactor creep rates of structural materials.

Effect of Simulated Wear in the Mechanical Properties of Unirradiated 20% CW 316 SS Prototypic Cladding

Stress rupture and transient heating tests were conducted on unirradiated 20% CW 316 SS tubing to determine the effect of simulated wear marks on mechanical properties. Data from these tests show that the effect of simulated wear marks can be evaluated by using the net-section stress. Results are summarized with the use of the Larson-Miller Parameter.

Low Temperature Creep of 20% CW 316 SS

Creep strains in 20% CW 316 SS at 850°F under a stress of 20,000 psi are small (i.e., a strain of 120×10^{-6} in 1000 hours), but readily measurable using the microwave extensometer. Creep strains measured during an in-reactor test were about seven times larger than those measured in the present laboratory experiment.

Effect of Neutron Fluence on the Mechanical Properties of 20% CW 316 SS

The effect of neutron fluence on the mechanical properties of 20% CW 316 SS tubing has been evaluated with a correlation based on the Larson-Miller Parameter and the Life Fraction Rule. Tensile strength, stress-rupture, burst and transient data were analyzed for neutron fluences of up to 2.65×10^{22} n/cm² ($E > 0.1$ MeV). The load-bearing capability has been reduced by about a factor of 2 for higher fluences.

Subcritical Crack Growth

The fatigue-crack propagation behavior of Incoloy 800 (ASME SB-409) in an air environment was studied at temperatures of 75, 600, 800, 1000 and 1200°F. Material in both Grades 1 and 2 was studied, and in general, the crack extension behavior of the two grades was similar over this temperature range. Fatigue-crack growth rates increased with increasing test temperatures, although the increases were not as great as observed in Types 304 and 316 SS tested under identical conditions. The improved behavior of Incoloy 800 is thought to be related to its superior oxidation resistance.

The fatigue-crack propagation behavior of SA-351, Grade CF8 cast stainless steel was investigated over the temperature range of 75 to 1200°F. Crack growth rates were slightly lower than in plate material tested under similar conditions. No effect of crack orientation was noted for the two orientations studied.

High Strain Rate Effects

The effect of thermal aging on the tensile properties and strain rate sensitivity of 304 SS has been determined. Specimens were thermally aged at temperatures of 800, 1000 and 1200°F for exposure times of 1000, 3500 and 10,000 hours. Testing was performed at the aging temperatures and at strain rates from 3×10^{-5} to sec^{-1} . Results of these tests indicate that thermal aging to 1200°F does not greatly alter the tensile properties or strain rate sensitivity of 304 SS.

FFTF/LMFBR Weldment Materials Evaluation

Pre- and post-irradiation creep-rupture results for CRE 308 SS weldment materials indicate irradiation-induced decreases in the minimum creep rates compensate to some extent for losses in creep ductility. The result is essentially no change in the rupture life thus indicating that, in terms of vessel lifetime, the effects of neutron irradiation will be negligible with regard to creep-rupture life considerations.

Tensile results for prototypic vessel weld deposits (CRE Type 308) following aging at 960°F for periods to 10,000 hours indicate strength increases and ductility losses with aging times from 0 to 3000 hours with only minor property changes beyond 3000 hours. These data are not in close agreement with results obtained at HEDL for an aging temperature of 1000°F or with those reported by ORNL for aging temperatures of 900 and 1100°F.

A pre- and post-irradiation tensile property evaluation has been conducted on prototypic FTR core basket base (304L) and weld (308) materials. Irradiation in the EBR II produced familiar increases in strength and reductions in ductility. However, estimates of projected tensile properties based on irradiation conditions which are potentially more severe than those expected in actual FTR service provide assurance of FTR core basket reliability overdue presently planned component lifetime.

Cladding Development and Process

The recovery and recrystallization characteristics of eleven prototypic and productive fuel cladding lots at 1400, 1500 and 1600°F have been determined to establish lot-to-lot variation and to provide a basis for possible improvement in cladding recrystallization characteristics. Recrystallization at these temperatures was found to be very sensitive to the heat treatment used just prior to the final 20% reduction in area.

Effects of Alloy Variations on Corrosion Behavior of Potential Cladding and Duct Materials

The effects of minor alloy variations on the corrosion of 316 SS have been investigated by exposure of specimens in the Source Term Control Loop (STCL). The results show that increases in the silicon and nickel content increase the corrosion rate, while increases in chromium tend to decrease the corrosion rate. Simultaneous variations in carbon and nitrogen were shown to have no effect on corrosion as determined by weight change measurements.

Long Replacement Sequences in Displacement Cascade Modeling

The approximate treatment of long replacement sequences was extended to 20 keV displacement cascades. The results were similar to those reported earlier at 2.5 and 5.0 keV. They suggest that total residual defect production in stainless steel is not grossly overestimated

by the currently recommended procedure for calculating displacements. Most of the interstitials, but only a small fraction of the vacancies, are mobile and hence escape from the cascade.

Damage Function Analysis of Cladding and Structural Materials

A draft has been generated of a RDT Standard "Adjustment of Fluence Limits for Materials by Use of the Energy-Dependent Damage Function." This standard is intended to define the procedures for applying damage functions used to predict the fluence required in a specific LMFBR spectrum to achieve a given property change.

The Predicted Effects of Experimental and Material Parameters on Void Nucleation

The current theory of homogeneous nucleation of voids in a supersaturation of vacancies and interstitials was utilized to predict the effect of various experimental and material parameters on such observable quantities as the void number density and the incubation period before the appearance of voids. It was found that both large, stable sink densities and decreased damage rates increase the incubation time. The surface energy and the bias term are also expected to have a large effect on the incubation period, with the surface energy affecting the nucleation rate and the bias term influencing the incubation time. The vacancy migration energy primarily affects the nucleation rate, resulting in a shift of the swelling to higher temperatures if the migration energy is increased. And finally, the lattice parameter has essentially no effect on either the nucleation rate or the incubation time, and therefore, will not influence the observed void number density or the incubation period.

Electron Beam Characterization for the JEM-1000-4 High Voltage Electron Microscope

Electron beam profiles have been measured as part of a continuing review and standardization of irradiation practices with the High Voltage Electron Microscope. Such precise definition of operating parameters is necessary to the conduct of quantitative electron irradiation experiments for simulation of neutron irradiation effects in duct and cladding alloys. Characterization of beam profiles was completed with the following results:

- 1) Little decrease in beam intensity (and therefore dose rate) results from decreasing beam spot size.
- 2) Little decrease in beam width results as the beam current is decreased.
- 3) Introduction of a 1000 μm condenser aperture decreases the beam intensity by as much as 55%, has little effect on beam diameter, and results in equal maximum intensities for all three beam spot sizes.
- 4) Beam Profiles can be accurately described by the Gaussian expression

$$I = I_0 \exp - (r^2/a^2)$$

where r is the distance from the beam center, I_0 is the maximum beam intensity and the parameter a describes the profile width.

Comparative Electron Irradiations of FTR First Core Tubing

A series of electron irradiations at 550 to 600°C is in progress on specimens cut from each of the various heats of FTR qualification and first-core fuel cladding tubes. Analysis

is completed for approximately 70% of the irradiations and shows a consistent set of conclusions. All specimens exhibit an incubation period followed by linear swelling behavior. The length of the incubation period varies strongly, however, reflecting a spot-to-spot inhomogeneity of void nucleation. This local variation is large enough so that heat-to-heat trends cannot be definitively discerned. Bands of deformation twins offer the greatest resistance to nucleation, but even these regions eventually yield to void formation.

The steady-state swelling rate which eventually develops appears to be independent of the void nucleation behavior, with the steady-state swelling rate at $\sim 580^{\circ}\text{C}$ and 19.3 dpa/hr constant at $1.4 \pm 0.2\%$ dpa for all lots studied. Irradiations at one third this displacement rate yield the same general behavior, but swelling rates of $1.0 \pm 0.2\%$ dpa. These and other data suggest that the difference between the results obtained at the two displacement rates is due to a stronger influence of beam heating than previously reported, requiring an adjustment in indicated irradiation temperatures.

Swelling of CW 316 SS Irradiated to High Neutron Fluences

Immersion density and preliminary transmission electron microscopy (TEM) examinations were completed on specimens of 20% CW 316 SS tubing irradiated in the unstressed condition to neutron fluences of about $9 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) in Subassembly X-098 in EBR-II. The maximum density decrease of 2.3% was measured for the 40 mg specimens irradiated at a calculated temperature of about 610°C . This specimen contained a duplex void size distribution, with the larger voids all associated with precipitates.

The swelling observed in these specimens is less than that predicted by the current Stress-Free Swelling Design Equation. Presuming a bilinear swelling behavior, this is probably due to a larger than predicted "incubation" fluence in the steel used in the X-098 experiment. Otherwise, the data do not show major inconsistencies with the current design equation.

Surface Effects in Irradiation Experiments

The influence of the foil surfaces in HVEM experiments appears to fall into two categories. The first effect is a restriction on void nucleation, which manifests itself in void-denuded zones adjacent to surfaces. Secondly, there are surface-related forces which can lead to the dissolution of voids whose surfaces protrude beyond the denuded zone boundary. Such surface perturbations can lead to a total suppression of swelling, the calculation of artificially low swelling rates or even saturation of swelling. Void loss appears to occur due to rapid shrinkage following a small amount of drift beyond the denuded zone boundary. In all cases, the void vanishes prior to intersection with the foil surface.

Aerojet Nuclear Company

FATIGUE AND CREEP-FATIGUE BEHAVIOR OF IRRADIATED AND UNIRRADIATED TYPE 304 AND 308 STAINLESS STEEL

G. E. Korth and M. D. Harper

I. IRRADIATION EFFECTS ON LOW CYCLE FATIGUE AND CREEP-FATIGUE OF 304/308 SS WELDMENTS

Specimens cut from SMA weldments with controlled residual elements were prepared from 2 3/8-in. thick FFTF residual plates and irradiated in EBR-II at 580 to 610°C (1076 to 1130°F) to fluences of $0.5-1.0 \times 10^{22}$ n/cm² ($E > 0.1$ MeV). The specimens were cycled in strain controlled, fully reversed tests at 593°C (1100°F) at a strain rate of 4×10^{-3} s⁻¹ with a closed loop electro-hydraulic system. Testing was conducted in air using induction heating to obtain the elevated temperature. Due to the anisotropy of the weld metal, specimens containing weld had a cylindrical gage section, and an axial extensometer with a 0.375-in. gage length was used. An hourglass specimen configuration was used with most base metal specimens. A few uniform gage base metal specimens were tested to verify correlation between the two geometries. Figure ANC-1 shows both specimen types. Further testing details can be found elsewhere.^(1,2)

A. FATIGUE BEHAVIOR AT 593°C (1100°F)

Unirradiated base and weld metal and irradiated weld material were reported in the previous semi-annual progress report in this series.⁽³⁾ One more irradiated weld specimen and three irradiated base metal specimens have been tested to complete the 593°C (1100°F) test matrix, and these additional specimens are reported in Table ANC-I, where

$\Delta \epsilon_t$ = total strain range	σ_t = tensile stress amplitude
$\Delta \epsilon_p$ = plastic strain range	N_f = cycles to failure
$\Delta \sigma$ = stress range	t_f = time to failure

and the entire test matrix is plotted in Fig. ANC-2. At the higher total strain ranges of 2% and 1%, the irradiated specimens exhibited cyclic lives of just about one-half that of the unirradiated weld metal; but at a total strain range of 0.5%, the irradiated material has cyclic lives of equal to or superior to the unirradiated material, and a crossover appears imminent. The relative positions of the irradiated data on Fig. ANC-2 of the various orientations have remained essentially the same as the unirradiated (i.e., weld metal specimens orientated parallel to the weld seam still have better fatigue resistance than transverse specimens and specimens with the fusion line in the gage section have the shortest fatigue lives). The irradiated base metal shows no significant reduction in fatigue life when compared to the unirradiated base metal. However, when a creep-fatigue condition exists, the results are quite different (see next section).

FIGURE ANC-1. Schematic Diagram Showing Specimen Details and Relative Positions of Specimens with Respect to Weldment Block.

FIGURE ANC-2. Effects of Irradiation on the Strain Fatigue Behavior of 308 SS Weld Metal at 593°C (1100°F) at a Strain Rate of $4 \times 10^{-3} s^{-1}$. Irradiated to Fluences of 0.3 to $1.0 \times 10^{22} n/cm^2$ ($E > 0.1$ MeV) at 580-610°C.

B. TENSILE HOLD TIME EFFECTS (CREEP-FATIGUE) OF SMA TYPE 304
/308 STAINLESS STEEL WELDMENTS AT 593°C

To investigate creep-fatigue interaction of the weld and base metal, tests were conducted with a hold period at the maximum tensile strain of each cycle. Since the strain is being held constant, stress relaxation occurs during the hold period as some of the elastic strain is converted to plastic strain. The irradiation history of the irradiated specimens is the same as given in the previous section. Tests were conducted with hold times of 0.01, 0.1 and 0.5 hours and compared to cyclic lives of tests without hold periods. Tabulated data of the irradiated base metal which had not been reported previously are shown in Table ANC-1. Figure ANC-3 is a

FIGURE ANC-3. Time to Fail vs Cycles to Fail for Irradiated & Unirradiated 308 Weld Metal Tested at 593°C (1100°F) at a Total Strain Range of 1.0%. Irradiated to Fluence Levels of 0.3 to 1.0×10^{22} n/cm² ($E > 0.1$ MeV) at 580-610°C.

time to fail, t_f , vs cycles to fail, N_f , plot of the hold time irradiated and unirradiated weldment tests conducted at a total strain range of 1% at 593°C (1100°F). This type of plot gives better resolution on the reduction of cyclic life as a function of hold time than a total strain range vs cycles-to-fail ($\Delta\epsilon_t - N_f$) plot and also allows extrapolations to longer hold periods.

Zero hold time or straight fatigue cycling data are shown for a reference line. The weld metal, which had a lower cycle life at one per cent strain range and zero hold time, was less damaged by the hold times than the base metal, and a crossover occurred at about a 0.1 hour

TABLE ANC-1

STRAIN FATIGUE BEHAVIOR OF 304/308 SS WELDMENTS TESTED^a AT 593°C (1100°F)

Specimen No.	Neutron Exposure ^b	Condition ^c	$\Delta\epsilon_t, \%$	$\Delta\epsilon_p, \%$	$\Delta\sigma$ at $N_f/2$		σ_t at $N_f/2$		σ_{tmin}^d at $N_f/2$		N_f	t_f, h	Hold time, h
					MPa	ksi	MPa	ksi	MPa	ksi			
BP-37	8.00	Base, 2a-P-C	1.80	1.33	815.0	118.2	386.8	56.1	-	-	788	1.92	0
BP-1	8.59	Base, 2a-P-S	1.01	0.63	653.6	94.8	313.7	45.5	-	-	2,841	3.88	0
BT-20	9.58	Base, 2a-T-S	0.41	0.12	496.4	72.0	282.0	40.9	-	-	42,127	22.97	0
D2C1	11.14	Weld, 2a-T-C	0.38	0.06	439.9	63.8	213.7	31.0	-	-	16,082	8.14	0
BT-29	2.71	Base, 2a-T-S	1.00	0.61	665.3	96.5	317.8	46.1	300.0	43.5	1,453	15.70	0.01
BP-7	4.73	Base, 2a-P-C	1.01	0.61	656.4	95.2	316.5	45.9	313.0	45.4	1,752	18.92	0.01
BT-21	5.15	Base, 2a-T-S	1.01	0.63	664.6	96.4	315.8	45.8	266.8	38.7	318	32.35	0.1
BP-27	12.19	Base, 2a-P-C	1.00	0.66	616.4	89.4	292.3	42.4	243.4	35.3	141	70.66	0.5
BP-29	4.99	Base, 2a-P-S	0.99	0.67	627.4	91.0	298.5	43.3	243.4	35.3	99	49.61	0.5

^aTriangular waveform with strain rate of $4 \times 10^{-3} s^{-1}$ ^bIrradiated to fluence shown in units of $10^{21} n/cm^2$ ($E > 0.1$ MeV) at 580-610°C^c2=Base metal heat 600414

a=As welded condition before irradiation

T=Specimen transverse to weld seam

S=Taken from section near surface of weldment

C=Taken from section near center of weldment

^d σ_{tmin} =tensile stress amplitude at end of hold period

hold time for the unirradiated material and closer to 0.01 hour for the irradiated condition. The parallel orientation again was seen to be superior to the traverse orientation. The depth in the weldment block had a much more pronounced effect in the hold time tests and, with few exceptions, the center section material had a longer life than the surface weld material. Considerably more scatter was introduced in the data due to irradiation, but, in general, its effect was to offset the cyclic life to lower values with about the same rate of life reduction as hold periods increased, except for the irradiated base metal, which degraded much faster with hold times longer than 0.01 hour hold. Compressive hold periods were not investigated in this work, but previous investigations for Type 304 and 316 SS base metal in the irradiated and unirradiated condition show this type of loading to be much less damaging than the tensile hold. (4,5)

The creep-fatigue data were somewhat surprising in that the effects of tensile hold periods were much less damaging in the weld metal than in the base metal. In another investigation⁽¹⁾, a particular heat of 304 SS exhibited superior creep-fatigue properties, much like the weld reported here, but that heat of SS was also reported to have superior creep properties. Creep studies of the unirradiated weld metal used in this investigation have shown that the weld material was equivalent, but not necessarily superior, to the base metal.⁽⁶⁾ The reason for the higher creep-fatigue resistance of the weld metal was probably due to the extensive cyclic softening experienced by the weld material. Even though the amount of stress relaxation during hold periods was approximately the same for base and weld metal, the mean tensile stress amplitude during a hold time was lower for the weld metal the major part of the cycle life. This behavior is illustrated in Figure ANC-4 for unirradiated material.

FIGURE ANC-4. Comparison of Tensile Stress Amplitude at Beginning and End of Hold Period of Base Metal as a Function of Cycle Life.

Irradiation resulted in significant decreases in the cycles to fail and introduced considerable scatter in the data. Irradiation probably interacted differently with the various species of the heterogeneous substructure resulting in early crack initiation in some cases and not in others. Helium bubbles from n, α reactions collecting at the grain boundaries or austenite-ferrite interfaces also undoubtedly played a significant role in the cyclic life when dwell periods could assist in their agglomeration. The marked creep-fatigue interaction of the irradiated base metal is not obvious, but probably is due to the difference in the way

that irradiation defects interact with the microstructure. The weld metal has a much more complex microstructure which could act as trapping centers for the irradiation defects, whereas these defects (especially helium) in the base metal would have a greater probability for agglomerating at grain boundaries, enhancing intergranular failure.

C. LOW CYCLE FATIGUE BEHAVIOR OF 304/308 SS WELDMENT MATERIAL AT 482°C (900°F)

Some of the unirradiated phase of the weldment material tested at 482°C (900°F) were reported previously,⁽³⁾ but since some tests were preliminary and no irradiated data were included, the entire test matrix is tabulated in Table ANC-2 and plotted in Fig. ANC-5. Test parameters and specimens are the same as described previously, except for test and irradiation temperatures.

FIGURE ANC-5. Strain Fatigue Behavior of 304/308 SS Weldment Material Tested at 482°C (900°F) at a Strain Range of 4×10^{-3} to 1. Irradiated to Fluences of 0.5 to 1.4×10^{21} n/cm² ($E > 0.1$ MeV) at 380 to 450°C.

The weld metal at this temperature is also inferior in fatigue properties when compared to base metal in the high strain range area, but approaches the base metal at lower strain ranges. The parallel orientation, however, appears to be equivalent to base metal from approximately 1% strain range and lower. With a neutron exposure of 0.5 to 1.4×10^{21} n/cm², $E > 0.1$ MeV, the fatigue life was not reduced as much by the irradiation as at the higher temperature and fluence, and the crossover point where the irradiated material became equivalent to the unirradiated material was at a higher strain range. In the high cycle region, the irradiated weld was equivalent to or slightly greater in fatigue resistance than the unirradiated base metal.

Two unirradiated base metal and one irradiated weld metal tests involving hold times were conducted at 1.0% strain range. No effect of hold time was seen at 482°C (900°F). Data for these specimens are included in Table ANC-2, but they are not plotted in Fig. ANC-5.

D. TENSILE TESTS OF WELDMENT MATERIAL

Results of limited tensile tests of irradiated and unirradiated weld metal are reported in Table ANC-3. The irradiated material increased in yield and ultimate strength and was reduced in ductility, as is normally observed with metals subjected to neutron radiation.

TABLE ANC-2

STRAIN FATIGUE BEHAVIOR OF 304/308 SS WELDMENTS TESTED^a AT 482°C (900°F)

Specimen No.	Condition ^b	$\Delta\epsilon_t, \%$	$\Delta\epsilon_p, \%$	$\Delta\sigma$ at First Cycle		$\Delta\sigma_{max}$		Cycle of $\Delta\sigma$ Max.	$\Delta\sigma$ at $N_f/2$		σ_t at $N_f/2$		$\Delta\sigma$ at $\sqrt{3}/4 N_f$		N_f	$t_{f,h}$
				MPa	ksi	MPa	ksi		MPa	ksi	MPa	ksi	MPa	ksi		
BT-100	Base 2a-T-S	2.02	1.53	356.5	51.7	833.6	120.9	137	799.8	116.0	384.7	55.8	789.5	114.5	685	1.85
BI-72	Base 2a-T-S	2.00	1.33	373.7	54.2	842.5	122.2	175	806.0	116.9	386.1	56.0	780.5	113.2	594	1.59
BT-2	Base 2i T-C	1.80	1.33	580.3	84.2	899.8	130.5	85	877.0	127.2	440.6	63.9	854.3	123.9	899	2.24
BT-23	Base 2a-T-C	0.99	0.59	393	57.0	605.4	87.8	379	555.0	80.5	281.3	40.8	533.0	77.3	3,809	5.20
BP-25	Base 2i P-C	0.89	0.57	410.9	59.6	641.9	93.1	155	571.6	82.9	278.5	40.4	568.1	82.4	5,477	6.74
BI-24	Base 2a-T-S	0.79	0.44	398.5	57.8	520.6	75.5	440	482.6	70.0	239.9	34.8	464.0	67.3	7,662	8.50
BI-25	Base 2a-T-S	0.50	0.21	322.7	46.8	---	---	---	416.4	60.4	208.2	30.2	---	---	28,176	19.60
BP-30	Base 2i P-S	0.39	0.10	366.8	53.2	568.8	82.5	265,000	510.9	74.1	231.7	33.6	538.5	78.1	265,000 ^f	143.8 ^f
BI-28	Base 2a-T-S	0.37	0.03	289.6	42.0	582.6	84.5	61,500	544.7	79.0	251.0	36.4	570.9	82.8	692,520	381.30
BI-22	Base 2a-T-C	0.33	0.01	---	---	---	---	---	359.2	52.1	164.8	23.9	---	---	984,324 ^f	467.10 ^f
WF-42	Weld 2a-FL-C	1.98	1.46	696.4	101.0	633.6	120.9	80	830.1	120.4	399.2	57.9	821.2	119.1	262	0.70
WI-58	Weld 2a-T-S	2.02	1.50	692.9	90.2	732.2	106.2	42	723.3	104.9	367.5	53.3	717.1	104.0	364	0.98
WI-26	Weld 2a-T-C	1.98	1.35	639.2	92.7	654.3	94.9	6	608.1	88.2	295.1	42.8	601.2	87.2	588	1.31
C2C6 (1.09)	Weld 2i-T-C	1.93	1.40	862.5	125.1	900.5	130.6	4	801.9	116.3	388.9	56.4	792.2	114.9	347	0.94
WP-76	Weld 2a-P-C	1.92	1.36	757.1	109.8	757.0	109.8	1	694.3	100.7	327.5	47.5	682.6	99.0	531	1.42
WP-21 (1.38)	Weld 2i-P-C	1.95	1.34	903.9	131.1	922.5	133.8	3	760.5	110.3	348.2	50.5	743.3	107.8	513	1.39
WF-57	Weld 2a-FL-S	0.98	0.55	599.9	87.0	644.0	93.4	50	612.9	88.9	313.7	45.5	590.9	85.7	1,583	2.21
WI-60	Weld 2a-T-S	1.01	0.57	---	---	619.2	89.8	160	599.2	86.9	293.7	42.6	585.4	84.9	1,042	1.43
WI-42	Weld 2a-T-C	1.00	0.54	744.6	108.0	744.6	108.0	1	641.2	93.0	303.4	44.0	622.6	90.3	1,434	1.96
D2B6 (1.07)	Weld 2i-T-S	0.99	0.53	775.7	112.5	782.6	113.5	10	686.7	99.6	337.2	48.9	667.4	96.8	875	1.19
WP-59	Weld 2a-P-S	0.98	0.50	497.8	72.2	538.5	78.1	93	487.5	70.7	232.4	33.7	470.9	68.3	4,094	5.60
WP-19 (1.42)	Weld 2i-P-S	0.99	0.47	688.1	99.8	688.1	99.8	1	580.5	84.2	286.1	41.5	566.8	82.2	4,821	6.58
WI-16 (0.94)	Weld 2i-T-S	0.80	0.36	775.0	112.4	775.0	112.4	1	664.7	96.4	312.3	45.3	650.2	94.3	2,022	2.21
WF-36	Weld 2a-FL-C	0.51	0.16	601.2	87.2	601.2	87.2	1	482.6	70.0	255.1	37.0	499.9	72.5	9,020	6.10
WI-22	Weld 2a-T-C	0.50	0.12	654.3	94.9	654.3	94.9	1	552.3	80.1	257.2	37.3	544.7	79.0	6,053	4.14
WP-79	Weld 2a-P-C	0.50	0.09	---	---	---	---	---	508.1	73.7	257.2	37.3	---	---	42,160	28.73
WI-59	Weld 2a-T-S	0.49	0.12	500.6	72.6	506.1	73.4	235	485.4	70.4	25.2	36.6	48.6	69.7	13,195	9.76
WI-29 (1.29)	Weld 2i-T-S	0.45	0.09	520.6	75.5	552.3	80.1	10	497.8	72.2	259.9	37.7	495.0	71.8	30,968	19.23
WI-56	Weld 2a-T-S	0.40	0.09	496.4	72.0	501.3	72.7	167	477.1	69.2	243.4	35.3	465.4	67.5	10,058	5.50
WI-46	Weld 2a-T-C	0.37	0.03	351.6	51.0	366.1	53.1	10	335.8	7	177.2	25.7	335.8	48.7	27,917	14.09
WP-77	Weld 2a-P-C	0.37	0.11	275.1	39.9	---	---	---	406.1	58.9	186.2	27.0	---	---	124,329	68.38
WP-70 (1.40)	Weld 2i-P-C	0.40	0.03	488.2	70.8	488.2	70.8	1	477.8	69.3	237.2	34.4	---	---	207,346 ^f	112.9 ^f
BT-26 ^c	Base 2a-T-C	0.98	0.62	---	---	---	---	---	553.7	80.3	273.7	39.7 ^e	---	---	3,818	43.4
BI-27 ^d	Base 2a-T-C	0.98	0.60	---	---	---	---	---	602.6	87.4	297.8	43.2 ^e	---	---	3,563	353.8
D2C7 ^d (1.30)	Weld 2i-T-C	1.00	0.56	866.0	125.6	866.0	125.6	1	721.9	104.7	339.9	49.3 ^e	707.8	102.7	1,050	106.4

^aTriangular waveform with strain rate of $4 \times 10^{-3} s^{-1}$ ^b2=Base metal heat 600414^aAs welded conditionⁱIrradiated to fluence shown in () in units of $10^{21} n/cm^2$ ($E > 0.1$ MeV at 380-450 C)^TSpecimen taken transverse to weld seam^PSpecimen taken parallel to weld seam^{FL}Transverse specimen across fusion line^SIsken from section near surface of weldment^CTaken from section near center of weldment^cTensile hold time of 0.01 hr on each cycle^dTensile hold time of 0.1 hr. on each cycle^eFirst number is tensile stress at beginning of hold period and second number in [] is tensile stress at end of hold^fDid not fail

Tensile tests of irradiated material were conducted only on transverse specimens due to the limited number of irradiated parallel specimens available.

A method has been proposed to predict the effects of irradiation on fatigue behavior when unirradiated fatigue data and irradiated and unirradiated tensile data are available.⁽⁷⁾ This method, called the Fraction Modification Method, is a fraction adjustment to both terms of the equation:

$$\Delta \epsilon_t = \Delta \epsilon_e + \Delta \epsilon_p = \phi_u A N_f^a + \phi_t B N_f^b$$

where $\Delta \epsilon_e$ is the elastic strain range, A, a, B, and b are constants determined from unirradiated fatigue data, and $\Delta \epsilon_t$, $\Delta \epsilon_p$ and N_f are the same as defined previously. The fraction modifications are ϕ_u and ϕ_t which are defined by:

$$\phi_u = \frac{(\sigma_{uts})_{\text{irrad.}}}{(\sigma_{uts})_{\text{unirrad.}}} \quad \phi_t = \left(\frac{D_{\text{irrad}}}{D_{\text{unirrad}}} \right)^{0.6}$$

where σ_{uts} is the ultimate tensile strength and D is fracture ductility $[= \ln(\frac{1}{1-RA})]$. RA is the reduction in area and the 0.6 exponent comes from the Universal Slopes Equation which is the basis for the Fraction Modification Method. Previous investigations^(4,7) have shown this method to be of value in predicting irradiation effects of fatigue data. The method was employed in this investigation and results are compared to the irradiated transverse weld metal in Fig. ANC-6. Sufficient irradiated parallel specimens were not available for tensile tests

FIGURE ANC-6. Comparisons of Irradiated Transverse 308 SS Weld Metal Fatigue Data to the Fraction Modification Predictions.

to allow comparisons with both orientations. Figure ANC-6 shows the prediction to be slightly nonconservative at the higher strain ranges where the ductility component of Eq 1 is dominant. This is probably due to the fact that the irradiated tensile data were obtained from specimens that were at the bottom of the fluence range at both temperatures. Tensile data from higher fluence specimens would have resulted in lower reduction-in-area values and thereby reduced the value of ϕ_t , which would bring the prediction curve down in the low cycle region.

TABLE ANC-3

TENSILE RESULTS OF 308 SS WELD METAL TESTED AT A STRAIN RATE OF $4 \times 10^{-3} \text{ s}^{-1}$

Specimen No	Condition ^a	Yield Strength		Ultimate Strength		Total Elong in 10 mm, %	Reduction in Area, %	Test Temperature Deg C
		MPa	ksi	MPa	ksi			
WT-49	Weld 2a-T-S	243.4	35.3	356.5	51.7	44	45	593
WP-61	Weld 2a-P-S	211.0	30.6	333.7	48.4	50	48	593
D2B9 (4.66)	Weld 2I-F-S	282.7	41.0	380.6	55.2	32	33	593
WI-57	Weld 2a-T-S	284.1	41.2	439.9	63.8	52	39	482
WI-34	Weld 2a-T-C	319.2	46.3	438.5	63.6	40	42	482
WP-80	Weld 2a-P-C	325.4	47.2	427.5	62.0	42	50	482
D2B5 (1.16)	Weld 2i-T-S	408.9	59.3	492.3	71.4	28	38	482
D2C4 (0.96)	Weld 2i-T-C	467.5	59.1	501.9	72.8	33	32	482

^a2=Base metal heat 600414

a=As welded condition

T=specimen taken transverse to weld seam

P=Specimen taken parallel to weld seam

FL=Transverse specimens across fusion line

S=Taken from section near surface of weldment

C=Taken from section near center of weldment

I=Irradiated to fluence shown in () in units of 10^{21} n/cm^2 ($E > 0.1 \text{ MeV}$) at 580-610 Ci=Irradiated to fluence shown in () in units of 10^{21} n/cm^2 ($E > 0.1 \text{ MeV}$) at 380-450 C

E. CONCLUSIONS OF WELDMENT FATIGUE TESTS

From the results of the low cycle fatigue tests with and without hold times on irradiated and unirradiated 308 SS weld metal, the following conclusions were drawn:

- 1) Irradiation to fluences of 0.5 to $1.1 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$ at 580 to 610°C resulted in reductions in fatigue life at 593°C when N_f was below about 5000 . However, in the higher cycle region, irradiated and unirradiated fatigue behavior were equivalent. At the lower test temperature (482°C) and specimens irradiated to 0.5 to $1.4 \times 10^{21} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$ at 380 to 450°C , fatigue behavior of the irradiated weld metal was only slightly inferior to the unirradiated material and then only in the low cycle region.
- 2) Changes in irradiated fatigue behavior could be primarily related to changes in strength and ductility, which allowed the Fraction Modification Method to be utilized in predicting irradiated properties.
- 3) Tensile hold times introduced into the fatigue cycling at 593°C produced less creep damage in the weld metal than in the parent material, which was attributed to the observation that the stress relaxation period of the weld occurred at a lower mean stress. Irradiation caused further reductions in the creep-fatigue interaction tests and the increased data scatter was attributed to inhomogeneous neutron damage of the various species of the weld substructure. Hold times had no significant effect on either base metal or irradiated weld metal at 482°C .

II. IRRADIATION EFFECTS ON FATIGUE BEHAVIOR OF CW SS

Work to characterize the effect of cold work on the fatigue properties of irradiated stainless steels is being investigated on a limited basis utilizing specimens on hand from previous irradiation programs. The purpose of this investigation is to determine if irradiation damage is more detrimental to cold worked material than to annealed. Three preliminary fatigue tests with hold times of 316 SS with 11% cold work irradiated and tested at 700°C indicated that the cold worked material was markedly inferior to the annealed material; however, in the unirradiated condition, the cold worked stainless steel was quite similar to the annealed material in cyclic behavior. These tests were conducted some months ago and other test commitments at that time did not allow pursuing the irradiation-cold work interaction further, but now the investigation will be continued even though a very limited number of irradiated specimens with cold work are available.

Two type 304 and two Type 316 irradiated specimens with 11% CW were fatigue-tested at 427°C (800°F) which corresponds approximately to the irradiation temperature. Unirradiated counterparts were also tested to establish a base line. The results of these tests are tabulated in Table ANC-4 and plotted in Figs. ANC-7 and ANC-8. Comparing the unirradiated CW data with unirradiated annealed data tested at the same temperature from various laboratories (ANL, GE, BMI) showed that the fatigue properties of both Type 304 and 316 11% CW to be slightly inferior to the annealed material. Since the irradiated 11% CW data is very close to the unirradiated, it can be concluded that at 427° (800°F) and low fluence (0.3 to $1.6 \times 10^{20} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$) cold work does not have a significant interaction with the irradiation damage. Tests are currently planned to investigate the cold work-irradiation damage interaction at higher temperature and fluence (700°C and $1.2 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$) with hold times where the preliminary tests previously mentioned indicated an area of concern.

TABLE ANC-4

STRAIN FATIGUE BEHAVIOR OF IRRADIATED AND UNIRRADIATED 304 AND 316 SS
WITH 11% COLD WORK TESTED^a AT 427°C (800°F)

Specimen No.	Specimen Type And Condition ^b	$\Delta\epsilon_t, \%$ @ $N_f/2$	$\Delta\epsilon_p, \%$ @ $N_f/2$	$\Delta\sigma$ @ $N_f/2$		σ_t max @ $N_f/2$		N_o^c	N_5^d	N_f	Time To Fail t_f, h
				MPa	ksi	MPa	ksi				
B-51	304 As Rec	1.52	1.08	746.0	108.2	359.2	52.1	1821	1828	1872	3.98
B-15	304 (.26)	1.52	1.10	789.5	114.5	388.2	56.3	1495	1511	1574	3.38
B-52	304 As Rec	0.80	0.46	582.6	84.5	275.1	39.9	9391	9401	9534	10.41
B-53	304 As Rec	0.51	0.21	593.6	86.1	270.3	39.2	-	-	29417	20.09
B-10	304 (15.7)	0.50	0.16	654.3	94.9	324.1	47.0	55505	55580	58280	39.63
E-51	316 As Rec	1.50	1.03	856.3	124.2	419.9	60.9	1808	1822	1868	3.95
E-9	316 (5.9)	1.50	1.00	988.0	143.3	484.0	70.2	1337	1375	1393	3.00
E-52	316 As Rec	0.81	0.40	726.0	105.3	356.5	51.7	5800	5889	6084	6.67
E-53	316 As Rec	0.51	0.14	485.4	70.4	255.8	37.1	27191	27282	29417	20.10
E-14	316 (0.72)	0.50	0.12	654.3	94.9	324.1	47.0	31459	31534	34759	23.71

^aTriangular waveform with strain rate of $4 \times 10^{-3} s^{-1}$

^bAs received or irradiated to fluence shown in () in units of $10^{20} n/cm^2$, $E > 0.1$ MeV at 380-450°C

^cNumber of cycles at crack initiation

^dNumber of cycles at 5% drop in tensile stress

FIGURE ANC-7. Strain Fatigue Behavior of Irradiated and Unirradiated 304 SS with 11% CW Tested at 427°C (800°F). Irradiated to Fluence Shown in () in Units of 10^{20} n/cm^2 , $E > 0.1 \text{ MeV}$, at 380 to 450°C.

FIGURE ANC-8. Strain Fatigue Behavior of Irradiated and Unirradiated 316 SS with 11% CW Tested at 427°C (800°F). Irradiated to Fluence Shown in () in Units of 10^{20} n/cm^2 , $E > 0.1 \text{ MeV}$, at 380 to 450°C.

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Atomics International

I. PROTON IRRADIATION OF 20% CW 316 SS

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The results of all irradiations are given in Table AI-1 and the dependence of swelling on displacement damage at irradiation temperatures of 500 and 600°C is shown in Figure AI-1. Swelling is here defined as $\Delta V/V_0$, where ΔV is the void volume and V_0 is the initial sample volume. At each temperature, the data are about equally well fitted by a linear expression and by a power law expression. Since there is little choice between the two, the dependence of swelling on displacements per atom (dpa) is given by the following two equations at each temperature:

FIGURE AI-1. Dependence of Swelling on Displacement Damage in Proton-Irradiated, 20% CW 316 SS.

$$\frac{\Delta V}{V_0} (\%) = -1.64 + 0.457 (\text{dpa}) \quad 500 \text{ C} \quad (1a)$$

$$\frac{\Delta V}{V_0} (\%) = 8.14 \times 10^{-3} (\text{dpa})^{2.35} \quad 500 \text{ C} \quad (1b)$$

$$\frac{\Delta V}{V_0} (\%) = -1.80 + 0.632 (\text{dpa}) \quad 600 \text{ C} \quad (2a)$$

$$\frac{\Delta V}{V_0} (\%) = 1.30 \times 10^{-2} (\text{dpa})^{2.37} \quad 600 \text{ C} \quad (2b)$$

The lines in Figure AI-1 are drawn according to the linear expressions, Equations (1a) and (2a).

The dependence of void density and average void diameter on displacement damage at 500 and 600°C are shown in Figures AI 2 and 3. At both temperatures, the void density rises rapidly at low damage levels, then reaches a plateau. The dependence of void diameter on number of displacements is essentially linear over a wide range of damage.

In Figures AI-1 and 2, the 500°C data at 2.2 and 9.6 dpa have been plotted as half-filled symbols. This was done to indicate that these data were obtained from TEM foils in which the atom displacement rates were the lowest (see Table AI-1). The results of void size, number density and swelling at 9.6 dpa (displacement rate 3.1×10^{-4} dpa/s). We assume, therefore, that the results obtained at 2.2 dpa were similarly unaffected by the low displacement rate. All data points were used in obtaining the least squares-fits - Equations (1a) and (1b).

In Figures AI-1 and 3, we have plotted the 600°C data at 4.3 and 8 dpa as half-filled symbols to indicate low displacement rates. It is clear from Figure AI-3 that the low displacement rates have resulted in unusually low void densities. This leads to low values of swelling in Figure 1. Thus, the data at 4.3 and 8 dpa have not been used in obtaining the dependence of swelling on damage - Equations (2a) and (2b).

Figures AI-4 and 5 illustrate void formation after high amounts of damage at 500°C and 600°C. Void agglomeration, involving two or three voids, has occurred at both temperatures, but at 500°C, very large voids, about 100 nm in size, were formed. Some of these are larger than the foil thickness and their appearance suggests that they also form by agglomeration. The density of these large voids is very low and they are responsible for about 10% of the total swelling.

Table AI-2 gives a comparison of TEM results between 20% CW 316 SS irradiated in X100 to a fluence of 4×10^{22} n/cm² ($E > 0.1$ MeV) and proton-irradiated samples. With respect to microstructure, there is agreement on loss of the CW structure during irradiation at 600°C and the presence of irradiation-induced precipitation at both 500°C to 600°C. The absence of helium bubbles after 600°C proton irradiation can be ascribed to the insertion of all the helium into the microstructure prior to irradiation. By the time carbides have formed, the pre-injected helium has apparently been incorporated into voids or other sinks.

Considering void formation in a general way, there are several points of qualitative agreement between the neutron and proton results. The most important of these are the inhomogeneity in void distribution within the CW microstructure, the reduction in swelling relative to that observed in solution-annealed-and-aged samples, and the lower swelling at 500°C

TABLE AI-1

VOID FORMATION IN 20% CW 316 SS

Irradiation Temperature (°C)	Proton Fluence (p/cm ²)	Damage (dpa)	Damage Rate (dpa/s)	Helium Content (appm)	Swelling (%)	Void Density (/cm ³)	Average Void Diameter (cm)	Sample Number
500	3.25×10^{19}	2.2	1.1×10^{-4}	5.9	0.031	9.9×10^{14}	0.77×10^{-6}	231-20
500	3.25×10^{19}	4.3	2.1	5.9	0.41	1.0×10^{16}	0.83	231-20
500	1.08×10^{20}	9.6	1.5	5.9	2.4	1.2×10^{16}	1.5	233-20
500	5.38×10^{19}	10	3.1	6.1	1.9	1.2×10^{16}	1.4	241-20
500	1.08×10^{20}	19	2.9	5.9	7.6	1.4×10^{16}	1.9	233-20
500	1.08×10^{20}	23	3.5	5.9	8.9	1.4×10^{16}	2.0	233-20
600	1.08×10^{19}	2.0	3.0	5.9	0.045	2.6×10^{14}	1.3	235-20
600	6.50×10^{19}	4.3	1.1	8.0	0.15	1.5×10^{14}	2.0	251-20
600	1.08×10^{20}	8.0	1.2	6.1	1.5	5.7×10^{14}	2.8	240-20
600	6.50×10^{19}	8.5	2.1	8.0	2.8	1.2×10^{15}	3.0	251-20
600	6.50×10^{19}	9.7	2.4	8.0	6.4	1.3×10^{15}	3.9	251-20
600	6.50×10^{19}	10	2.5	8.0	3.1	1.5×10^{15}	3.0	251-20
600	1.08×10^{20}	16	2.5	6.1	7.5	1.6×10^{15}	4.0	240-20
600	1.08×10^{20}	24	3.6	6.1	14	1.6×10^{15}	5.0	240-20

TABLE AI-2

COMPARISON OF NEUTRON AND PROTON-IRRADIATED 20% CW 316 SS

<u>NEUTRON</u> <u>500C</u>	<u>PROTON</u> <u>500C</u>
<ul style="list-style-type: none"> -Inhomogeneous void distribution related to cold-worked microstructure. -No detectable loss of cold-work structure. -Irradiation-induced $M_{23}C_6$. -Swelling less than in solution-annealed-and-aged samples due to a reduced void size and density. -At $\Delta V/V_o = 0.01\%$, $\rho_v = 4 \times 10^{13} \text{ cm}^{-3}$ and $d_{ave} = 1.5 \times 10^{-6} \text{ cm}$. 	<ul style="list-style-type: none"> -Inhomogeneous void distribution related to cold-worked microstructure. -No detectable loss of cold-work structure. -Irradiation-induced $M_{23}C_6$ (probable). -Swelling less than in solution-annealed-and-aged samples due to a reduced void size. Void density higher in cold-worked samples. -At $\Delta V/V_o = 0.03\%$, $\rho_v = 9.9 \times 10^{14} \text{ cm}^{-3}$ and $d_{ave} = 0.77 \times 10^{-6} \text{ cm}$.
<u>600C</u>	<u>600C</u>
<ul style="list-style-type: none"> -Inhomogeneous void distribution related to cold-worked microstructure. -Significant loss of cold-work structure. -More $M_{23}C_6$ than at 500C. -Swelling greater than at 500C due to increase in void size. Void density lower at 600C. -Swelling less than in solution-annealed-and-aged samples due to a reduced void size. Void density the same. -Some small helium bubbles at carbide-matrix interfaces. -At $\Delta V/V_o = 0.08\%$, $\rho_v = 2 \times 10^{13} \text{ cm}^{-3}$ and $d_{ave} = 3.2 \times 10^{-6} \text{ cm}$. 	<ul style="list-style-type: none"> -Inhomogeneous void distribution related to cold-worked microstructure. -Significant loss of cold-work structure. -More $M_{23}C_6$ than at 500C. -Swelling greater than at 500C due to increase in void size. Void density lower at 600C. -Swelling less than in solution-annealed-and-aged samples due to a reduced void size. Void density higher in cold-worked samples. -No helium bubbles. -At $\Delta V/V_o = 0.15\%$, $\rho_v = 1.5 \times 10^{14} \text{ cm}^{-3}$ and $d_{ave} = 2.0 \times 10^{-6} \text{ cm}$.

FIGURE AI-2. Dependence of Void Density and Average Void Diameter on Displacement Damage at 500°C in Proton-Irradiated, 20% CW 316 SS.

FIGURE AI-3. Dependence of Void Density and Average Void Diameter on Displacement Damage 600°C in Proton-Irradiated, 20% CW 316 SS.

FIGURE AI-4a. Void Formation in 316 SS, 20% CW Proton-Irradiated at 500°C to 23 d/a Seen at Low Magnification.

FIGURE AI-4b. Void Formation in 316 SS, 20% CW, Proton-Irradiated at 600°C to 40 to 23 d/a Seen at High Magnification.

FIGURE AI-5a. Void Formation in 316 SS, 20% CW, Proton-Irradiated at 600PC to 24 d/a Seen

FIGURE AI-5b. Void Formation in 316 SS, 20% CW, Proton-Irradiated at 600°C to 24 d/a Seen at High Magnification.

AI-10

FIGURE AI-6a Microstructure of 405 SS Alpha-Annealed Irradiated in FRR-II at 500°C to 4×10^{22}

AI-11

FIGURE AI-6b. Microstructure of 405 SS, Alpha-Annealed, Irradiated in EBR-II at 600°C to 4×10^{22} n/cm² (E>0.1 MeV)--Low Magnification.

FIGURE AI-7a. Microstructure of 405 SS, Alpha-Annealed, Irradiated in EBR-II at 600°C to 4×10^{22} n/cm² ($E > 0.1$ MeV)--Low Magnification

FIGURE AI-7b. Microstructure of 405 SS, Alpha-Annealed, Irradiated in EBR-II at 600PC to 4×10^{22} n/cm² (E>0.1 MeV)--High Magnification.

than at 600°C. There are obvious differences in the results, particularly with reference to void density. However, there are differences of this type even between neutron-irradiation results.

In the course of a 600°C proton irradiation, a pronounced loss of the CW structure occurs. The loss of the CW structure may be much less after neutron irradiation at 500°C, even at high damage levels. Thus, at high damage levels, comparison of swelling may be between two entirely different microstructures. More neutron data must be obtained before we can be assured of the efficacy of comparing swelling in CW SS created by proton (or any charged particle) irradiation with that created by neutrons. Only then can we be confident that simulation experiments can be used to extrapolate to higher damage levels the swelling obtained in reactor-irradiated metals.

II. TEM OF NEUTRON-IRRADIATED SS

A. G. Pard, K. R. Garr and D. Kramer

TEM has been performed on 405 SS irradiated in X100 at 500°C and 600°C to a fluence of 4×10^{22} n/cm² ($E > 0.1$ MeV). Prior to irradiation, the samples were annealed at 760°C for 24 hours and therefore were completely alpha phase with some carbides. Only a few voids were found at either temperature.

A high density of defect clusters formed at 500°C with a denuded zone adjacent to grain boundaries (Figure AI-6). Some fine precipitates are also present. At 600°C, these features are absent but some helium bubbles, about 15 nm in size, were located on grain boundaries and carbide-matrix interfaces (Figure AI-7).

General Electric Company

STRUCTURAL MATERIALS IRRADIATIONS

H. Busboom

This series of tests will provide design base data concerning the effects of irradiation on the mechanical and physical properties of core structural materials. The tests have been designed to complement ongoing and planned irradiation programs conducted by other contractors.

I. SERIES L-16 CAPSULES (EBR-II S/A X098A)

The L-16 capsules are designed to identify the temperature-dependence of alloy swelling by irradiating protoypical fuel cladding tubing at various temperatures and monitoring the growth of these tubes as a function of fluence. Subassembly X098A, which contains 20% CW 316 SS at $\sim 8 \times 10^{22}$ n/cm ($E > 0.1$ MeV), was discharged from EBR-II following Run 67. The interim examination has been completed at ANL-HFEF in Idaho, and selected materials have been re-encapsulated for continued irradiation starting with Run 71. The results from the interim examination are presented below.

A. MATERIAL

Both 304 and 316 SS were irradiated in S/A X098A similar to those materials irradiated in S/A X098 and reported earlier. Additional capsules containing 321 SS were added to S/A X098A; these data will be reported in later reports.

B. IRRADIATION HISTORY

The irradiation history for subassembly X098A summarized in Table GE-1 is similar to the history for S/A X098 summarized earlier⁽¹⁾. It is presently thought that the gamma heating rate for these capsules is lower than that reported earlier; the best estimated of the temperatures of irradiation for specimens in both S/A X098A and X098 are shown in Table GE-1.

C. POST-IRRADIATION EXAMINATION

The post-irradiation examination was primarily concerned with the dimensional changes of the specimen inserts. O.D. measurements were made on all specimens using a mechanical profilometer. These measurements were averaged to determine a mean diameter near the center of each specimen. The differences between pre- and post-irradiation diameter were used to calculate volume swelling assuming isotropic swelling behavior, i.e., $\Delta V/V = 3\Delta D/D$. The uncertainty in these measurements was estimated to be ± 0.0002 inches. The uncertainty for the reported swellings was estimated to be $\pm 0.3\%$.

The results for the core specimens are shown in Table GE-2 for both S/A X098A and the earlier S/A X098. Table GE-2 shows that all materials exhibited measurable swelling ($> 0.3\%$) at one or more temperatures when irradiated to $\sim 7.8 \times 10^{22}$ n/cm², whereas at 3.2×10^{22} n/cm², the only specimens which showed volume increases were solution-annealed 304 SS and specimens given aging treatments^(2,3) to induce carbide precipitation prior to irradiation.

TABLE GE-1
EBR-II HISTORY FOR S/A X098A CAPSULES

EBR-II Runs	56 to 67
Core Position	4A3
MWD's Total	
EBR-II	30,155
GE Burn-II	27,441
MWD's @ 62.5 MW (MWD's)	26,260
Time @ 62.5 MW (hr)	10,080
Average Peak Fluence >0.1 MeV(n/cm ²) ⁽¹⁾	8.58 x 10 ²²
Average temperatures ⁽²⁾ of core specimens in both S/A X098 and S/A X098A:	
286E240-G1	670 (~1240°F)
286E240-G2	580 (~1080°F)
286E240-G3	515 (960°F)
286E240-G3	450 (840°F)

1. Fluence based on 2.95×10^{18} n/cm²/MWD for E>0.1 MeV as calculated by BURN-II.
2. Temperatures based on 0.91 of peak gamma heating rate which was assumed to be 4.2 W/gm for both S/A's.

TABLE GE-2
RESULTS FOR L-16 PRIMARY SPECIMEN'S SWELLING⁽¹⁾

	450°C		525°C		600°C		675°C	
	3.2	7.8	3.2	7.8	3.2	7.8	3.2	7.8
<u>304 SS</u>								
SA	1.56	6.20	0.48	3.50	0.12	1.80	0.12	0.12
Carbide Agglomerated ⁽⁴⁾	1.80	--	0.60	--	0.18 ⁽³⁾	--	0	--
10% CW	--	4.20	--	5.20	0	0.12	--	0
20% CW	-0.12	2.80	0.24	4.30	-0.12	0	0	0
30% CW	--	1.80	--	0.96	-0.24	0	--	0.24
50% CW	--	--	--	--	--	0.60	--	--
<u>316 SS</u>								
SA	-0.12	1.40	-0.12	0.80	0.12	4.00	0	0.36
Carbide Agglomerated ⁽⁴⁾	0	--	0	--	0.60 ⁽³⁾	--	0.36	--
10% CW	--	-0.48	--	0.24	-0.12	3.10	--	0
20% CW	-0.24	-0.48	0	0.48	0.24	1.72	-0.24	-0.12
30% CW	--	0	--	0.24	-0.12	0.48	--	-0.36
50% CW	0	--	--	--	-0.18 ⁽³⁾	--	-0.12	--
Garofalo ⁽⁵⁾	0.60	--	0.72	--	0.54	--	0.24	--

- 1) Swelling is in volume percent, uncertainty in values estimated to be $\pm 0.3\%$.
- 2) Fluence is average for core regions and ~0.91 S/A average peak fluence in 10^{22} n/cm² (E>0.1 MeV).
- 3) Average of two specimens.
- 4) Carbide agglomerated heat treatment consisted of aging the SA material at 900°C for 24 hours (Ref 3).
- 5) Garofalo heat treatment consisted of aging the 30% CW material at 842°C for 24 hours followed by 704°C for 216 hours (Ref 2).

The largest swelling was observed in solution-annealed materials. Maximum values were 6.2% for 304 and 4.0% for 316 SS. Cold work effectively reduced the swelling for 316 SS, and the higher the level of cold work, the less was the observed swelling (See Figure GE-1). The maximum swelling measured for 20% CW 316 SS was 1.72%, or less than half that observed for 316 SS in the annealed condition. Cold work was less effective in reducing swelling of 304 SS (See Figure GE-1).

Although the peak swelling temperatures for these stainless steels could not be precisely determined from these L-16 data, it is apparent from Figure GE-1 that the peak swelling temperature for 304 SS is much lower than that for 316 SS. The peak for Type 304 shown in Figure GE-1 falls below 525°C, whereas the peak for Type 316 is shown at 600°C, well above 525°C.

The annealed Type 304 data from S/A X098A are in excellent agreement with similar measurements taken from capsule tubes containing GE F2-series fuel pins⁽⁴⁾ irradiated to approximately the same fluence. These earlier measurements show a peak swelling of 7% in the temperature range 450 to 470°C. This similarity adds further support to the most recent temperature estimates shown in Table GE-1.

Cold work apparently raises the peak swelling temperature slightly for 304 SS at the fluence level attained to date of $\sim 8 \times 10^{22} \text{ n/cm}^2 \text{ E} > 0.1 \text{ MeV}$. This increase was not observed for 316 SS. The swelling peak shown in Figure GE-1 is in general agreement with that observed by U.K. workers⁽⁵⁾ for annealed and CW M 316 irradiated in DFR. The DFR data show that peak swelling also occurs at $\sim 600^\circ\text{C}$ for these materials.

Prior to re-encapsulation for continued irradiation, small disks were extracted from the 20% CW 316 SS specimens. The specimens are being examined by transmission electron microscopy (TEM) to complement the profilometry data.

II. SWELLING OF SODIUM EXPOSED MATERIALS

As part of the GE swelling program, materials previously exposed to sodium were irradiated in EBR-II to determine the effects of prior sodium exposure on alloy swelling behavior. The three alloys investigated were Types 316, 347 and 321 SS in the SA condition. All materials were irradiated as 1/2-inch or 1-inch long rings cut from 0.25-inch O.D. x 0.015-inch wall tubing which was previously exposed unstressed to 704°C sodium for either 10,000 hours or 15,000 hours. Detailed characterization of these materials was reported previously.⁽⁶⁾ Archive samples (as-received tubing from vendor) were included in the irradiation to provide baseline swelling values for specimens not exposed to sodium.

The irradiation vehicle was the Mark A-19 capsule designated LP4C which was designed to irradiate the ring samples at $\sim 500^\circ\text{C}$. The rings were held concentrically on a mandrel inside the capsule tube and temperature was maintained by varying the amount of material inside the mandrel to account for the axial variation in gamma heating rate. A peak fluence of $5.2 \times 10^{22} \text{ n/cm}^2 \text{ E} > 0.1 \text{ MeV}$ was accumulated on the capsule in S/A X098A of EBR-II.

Immersion density measurements were taken before and after irradiation. The results, expressed as changes in density, are shown in Table GE-3. Clearly, the sodium exposure has enhanced alloy swelling in all three materials. It is quite remarkable that such large swellings (of up to 16%) were obtained in the sodium exposed materials for the rather low fluences

FIGURE GE-1. Swelling of 304 and 316 Tubing in EBR-II at Fluences of 7.0 to 8.5×10^{22} n/cm^2 ($E > 0.1$ MeV).

($\sim 5 \times 10^{22}$ n/cm² E > 0.1 MeV), whereas the unexposed materials exhibited swellings <2% for similar irradiation conditions. Also evident from these data is the much lower swellings for the sodium exposed Type 321 (<9%) as compared to the exposed Types 347 (16%) and 316 (13%).

TABLE GE-3
EFFECT OF SODIUM EXPOSURE ON THE SWELLING OF AUSTENITIC STAINLESS STEELS

Alloy	Sodium ⁽¹⁾ Exposure (hr)	Distance from Flux Midplane (in.)	Irradiation Conditions		$\Delta P/\rho_0$ (%)
			Fluence ⁽²⁾ (10^{22} n/cm ²)	Temp (°C)	
Type-347	None	-1.6	5.02	480	-1.95
Type-347	10,000	4.0	4.45	475	-11.68
Type-347	10,000	-3.6	4.55	480	-10.17
Type-347	15,000	5.5	3.93	500	-6.83
Type-347	15,000	-0.8	5.12	485	-16.19
Type-321	None	2.0	4.97	480	-1.12
Type-321	10,000	-4.6	4.30	490	-3.71
Type-321	10,000	3.0	4.76	480	-4.73
Type-321	15,000	5.7	3.88	470	-3.87
Type-321	15,000	-0.3	5.18	490	-8.52
Type-316	None	1.0	5.12	485	-0.73
Type-316	10,000	5.0	4.14	470	-8.65
Type-316	10,000	-2.6	4.86	480	-11.03
Type-316	15,000	6.2	3.62	475	-6.15
Type-316	15,000	0.2	5.18	490	-13.24

(1) In flowing sodium loops at 704°C.⁽¹⁾

(2) E>0.1 MeV

Although both temperature and fluence varied axially and therefore varied from specimen to specimen, it is believed that the swellings shown in Table GE-3 are more dependent on fluence than temperature, since tapered insert compensators were used to provide a reasonable constant heating rate in the core region of the capsule. Therefore, the swellings are plotted against fluence on linear scales assuming temperature to be constant (see Figure GE-2). As shown, a single straight line could represent either the 316 or the 347 SS data, indicating that little, if any, differences exist in the swelling behavior of these alloys after exposure in sodium for 10,000 or 15,000 hours. However, the data for Type 321 suggest that more swelling occurs in samples exposed for 15,000 hours than in those exposed for 10,000 hours.

The most important feature in Figure GE-2 is the steep slope indicated by all data sets, which suggests that not only is the amount of swelling very extensive for sodium exposed materials, but also the swelling rate is very high. For the Type 347 data as shown, about 50% swelling can be projected at a fluence of 10^{23} n/cm² E > 0.1 MeV.

FIGURE GE-2. Effect of 704°C Sodium Exposure on Alloy Swelling of Austenitic Stainless Steels. Temperature of Irradiation was $\sim 480^\circ\text{C}$.

The compositional and microstructural changes which occurred in these tubing specimens as a result of the 704°C exposure to flowing sodium have been described previously.⁽⁶⁾ Ongoing reactor irradiation tests at ORNL and HEDL, together with nickel ion bombardment tests at GE, show that carbon and nitrogen in solid solution play a large role in determining the extent of swelling in stainless steels. Further work is necessary to determine whether depletion of these elements from the alloy matrix by either precipitation or leaching during sodium exposure is indeed the prime reason for the enhanced swelling measured in the specimens reported here. It is clear, however, that neither major variations in the base alloy chemistry or externally applied stress are required to produce anomalously large swellings in austenitic stainless steels, since the bulk composition remained constant during sodium exposure of the Type 347 specimens and none of the samples were stressed during either sodium or neutron exposures.

Analysis is proceeding to assess the impact of these results in two areas of importance to LMFBR core performance. First, while the results given here are from an "extreme case" test in which clad specimens previously exposed for up to 15,000 hours at 704°C were then irradiated at ~500°C, it is nevertheless likely that some enhanced swelling will occur during simultaneous exposure to sodium and neutron flux or during some proposed fuel bundle shuffling schemes. Second, present projections of swelling and irradiation creep in 20% CW 316 SS for use in core design were developed assuming that enhanced swellings observed in 304 SS EBR-II driver fuel clads were due solely to stress effects. These design projections for CW 316 are consequently highly stress-dependent and are likely to be in error, since compositional changes would have undoubtedly occurred in the sodium-bonded driver fuel clads.

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Hanford Engineering Development Laboratory

I. IRRADIATION DAMAGE TO REACTOR METALS

T. T. Claudson

A. SENSITIVITY ANALYSIS OF HISTORY EFFECTS EXPERIMENT H-3

G. L. Wire/J. L. Straalsund, J. J. Holmes

1. Objective

Present plans call for irradiating an experiment to determine the effects of prior irradiation history on irradiation creep. The experiment consists of a series of different types of relaxation specimens and one type of primary loaded-beam specimen. The irradiation creep will be studied through a series of interim examinations on these specimens. The objective of the present work was to establish whether the sensitivity of the planned specimen designs will be adequate to attain the goals of the experiment. In this work, only the main sources of experimental uncertainty (which are stress measurements and specimen geometry changes),

2. Accomplishments and Status

In this Section, the sensitivity analysis is given for a stress relaxation test on 20% CW 316 SS which incorporates a slit tube sample configuration. In the next three Sections, analyses are presented for experiments on 304 SS which are designed to test the relationship of creep and swelling properties under various loading conditions.

3. Slit Tube Stress Relaxation

The goal of this experiment is to measure the change in irradiation-induced deformation rates as a function of neutron fluence for prototypic 20% CW 316 SS which has been pre-irradiated to fluences in a range of 0 to 3.8×10^{22} n/cm² ($E > 0.1$ MeV). Although a region of transient deformation may occur in the early part of the irradiation (as in the XX03 experiment), steady-state creep is initially assumed to illustrate the magnitude of measurement errors. For steady-state creep, the creep coefficients can be determined by measuring the stress relaxation time, t_r , due to irradiation-induced deformation. In the case of irradiation-induced deformation, the relaxation time t_r will ultimately be expressed in terms of neutron fluence in the same way as are irradiation creep coefficients, but for convenience, the term "relaxation time" is used here.

For steady-state creep at constant stress, the strain rate is given as:

$$\dot{\epsilon} = B\sigma \quad (1)$$

where σ is the stress and B is the creep coefficient. In stress relaxation under uniaxial load with deformation determined by Eq. 1, the stress will be reduced as a function of time as:

$$\sigma = \sigma_0 e^{-t/t_r}$$

where σ_0 is the initial stress and t_r is:

$$t_r = 1/BE \quad (2)$$

where E is Young's modulus. Calculations follow this report for the case of the loaded slit tube and which show that a similar relationship holds between the creep coefficient and the relaxation time so that the creep rate B can be determined from the rate of stress relaxation in this case as well.

The main uncertainty in determining the creep rate B will be due to the uncertainty in the stress measurement. The residual stress measurement technique developed for this experiment is schematically illustrated in Figure HEDL-1. This technique, conceptually the same as the so-called Manjoine technique, allows determination of the residual stress by measuring the force required to pry the slit tube open from its mounting block. The observed uncertainty in this lift-off force has experimentally been determined to be ± 0.05 lb. The maximum fiber stress is related to this force as:

$$\sigma = \frac{6082 P}{\ell} \quad (\text{Ref. 1}) \quad (3)$$

where P is the force and ℓ is the tube length, so that for a 0.75-inch specimen, the uncertainty in stress is of the order ± 400 psi. The upper curve in Figure HEDL-2 is an anticipated stress relaxation curve based on Eq. 1 for in-reactor deformation of 20% CW 316 SS and includes the uncertainty estimates for two initial stress levels. The lower curve has been included to illustrate the effect of a possible transient region of deformation such as observed in the XX03 test on uniaxial specimens. It is seen that this technique would allow ready detection of such a transient.

The steady-state creep rate may be determined by comparing two points on the stress relaxation curve (in the steady-state regime, of course). The percent error in the creep coefficient determined in this way is a function of the two stress levels chosen and is given by:

$$\frac{\Delta B}{B} = \pm \frac{\Delta\sigma/\sigma_1 \pm \Delta\sigma/\sigma_2}{\ln \sigma_2/\sigma_1} \quad (4)$$

For a high initial stress, we may have $\sigma_2 = 40,000$ psi, $\sigma_1 = 20,000$ psi giving $\Delta B/B = \pm 4.3\%$; while for a low initial stress $\sigma_2 = 10,000$ psi, $\sigma_1 = 5,000$ psi giving $\Delta B/B = 17.3\%$.

4. Bent Beam Stress Relaxation

This experiment is designed to test the dependence of creep rate on swelling by observing the stress relaxation rates of specimens cut from 304 SS duct. The specimens will be chosen to cover a range of prior irradiation of 0 to 10^{23} n/cm² ($E > 0.1$ MeV). The specimens will be stressed in a mandrel bending device, as shown in Figure HEDL-3, and measurements of the residual stress will be made to determine the creep rates in the same general way as described above for slit tubes. In this case, however, the residual stress will be determined from the changes in the radius of curvature of the specimens as they plastically relax. The residual stress (on the outer-most fiber of the bent beam) is given as:

$$\sigma = \frac{Ed}{2} (1/R_m - 1/R) \quad (5)$$

A. LOADED SLIT TUBE

B. MEASUREMENT

C. RESULTS

HEDL 7312-40.3

FIGURE HEDL-1. Slit Tube Stress Relaxation.

FIGURE HEDL-2. Stress Relaxation Versus Additional Fluence.

A MANDREL LOADING

B. MEASURING DEVICE

C. RESULTS: RESIDUAL STRESS DETERMINED

$$\sigma \propto \left(\frac{1}{R_s} - \frac{1}{R_m} \right)$$

R_s = SPECIMEN RADIUS OF CURVATURE

R_m = MANDREL RADIUS OF CURVATURE

HEDL 7312-40.4

FIGURE HEDL-3. Bent Beam Stress Relaxation.

where d is the beam thickness, R_m is the mandrel radius of curvature, and R is the relaxed specimen radius of curvature. The specimen radius of curvature will be determined by tracing the profile of a bent beam using two LVDT's, as shown in Figure HEDL-3. The results of such a measurement are shown in Figure HEDL-4. The error in the measurement of R will be due to the error ΔY in the vertical LVDT, as the bowing deflection Y for these beams is of order 0.020 inch and in terms of this quantity:

$$\Delta \sigma = \pm \frac{16 E D \Delta Y}{\ell^2} \quad (6)$$

where ℓ is the specimen length. For $\ell = 2$ inches, and $d = 0.040$ inch, and the observed of $\Delta Y = 1.3 \times 10^{-4}$ inches, the error in stress is ± 520 psi. The percent error in the creep coefficient for $\sigma_2 = 20,000$ psi, $\sigma_1 = 10,000$ psi is $\Delta B/B = \pm 11.4\%$.

According to current interpretation, the creep rate is enhanced by swelling, so that for the case of the bent beams in steady-state creep:

$$\dot{\epsilon} = [B + (D + P/9) S] \sigma + S/3 \quad (7)$$

FIGURE HEDL-4. Mandrel Beam Measurements.

where $\dot{S}$ is the swelling rate and D and P are the swelling enhancement coefficients. If this formulation is correct, then the relaxation time in the presence of swelling:

$$t_r = \frac{1}{E [B + (D + P/9) \dot{S}]} \quad (8)$$

will then be a function of swelling rate, and the higher rate swelling specimens will relax at a greater rate. In order to see what effects the swelling enhanced creep rate would have on a stress relaxation curve, nominal values for the coefficients in Eq. 8 were used to construct Figure HEDL-5 which shows the relaxation expected after a fluence of 0.4×10^{22} n/cm² ($E > 0.1$ MeV) as a function of swelling. For this graph, the values of $D = 2 \times 10^{-5}$ /psi, $P = 10^{-4}$ /psi, and $B = 3.3 \times 10^{-30}$ (n/cm² psi) were used. The swelling was taken to have the form $\Delta V/V \propto (\phi t)^2$ with the swelling rate of $1\%/(10^{22}$ n/cm²) at a fluence of 10^{23} n/cm²; which is a simplified approximate version of the empirical relationship deduced by Bates and Straalsund.⁽²⁾ These parameters give a swelling-enhanced creep coefficient of $(1.1 \times 10^{-29}/(\text{n/cm}^2 \cdot \text{psi}))$ at a fluence of 2.5×10^{22} n/cm², which is in reasonable accord with Walter's data.⁽³⁾

As can be seen from Figure HEDL-5, the amount of stress relaxation for this fluence is a strong function of the swelling, the stress dropping to $0.55 \sigma_0$ at 0.2% swelling and to $0.04 \sigma_0$ at 5% swelling. The dashed line in the figure represents the situation where the creep rate is independent of swelling ($D, P = 0$) and it is clear that a definite choice can be made between these possibilities unhindered by experimental error.

FIGURE HEDL-5. Stress Relaxation Versus Prior Swelling.

HEDL 7401-65.1

FIGURE HEDL-6. Swelling Driven Creep.

5. Swelling-Driven Creep

This experiment consists of a composite beam made up of a high swelling 304 SS member rigidly attached at its ends to a fresh 304 SS beam of the same cross section, as shown in Figure HEDL-6. Based on Eq. 7, it can be shown that the swelling beam will, after a sufficiently long time, produce final stresses on each specimen of magnitude:

$$\sigma = \frac{\dot{S}}{3 [2B + (D + P/9) \dot{S}]} \quad (9)$$

where $\dot{S}$ is the swelling rate of the high swelling member and the fresh member has been assumed to have a negligible swelling rate. The stress σ is a measure of the ratio of swelling rate to creep rate, and it will be measured by allowing the composite to bow to its equilibrium configuration. The stress in terms of the resulting radius of curvature R is calculated to be:

$$\sigma = \frac{2Ed}{3R} \quad (10)$$

where d is the specimen thickness. The error in σ is :

$$\Delta\sigma = \frac{+ 32Ed}{3\ell^2} \Delta Y \quad (11)$$

where ΔY is the same as in Section B as the same instrument will be used. The error in stress is then:

$$\Delta\sigma = \pm 350 \text{ psi} \quad (12)$$

Figure HEDL-7 shows the final stress σ as a function of the creep swelling coefficients $D + P/9$ using $B = 3.3 \times 10^{-30}/(\text{psi n/cm}^2)$ and $\dot{S} = 10^{-24}/\text{n/cm}^2$ in Eq. 9. It is seen that for the commonly assumed values of $D = 2 \times 10^{-5}/\text{psi}$, $P = 1 \times 10^{-4}/\text{psi}$ the residual swelling-induced stress is expected to be 10,000 psi; while for $D = P = 0$, $\sigma = 50,000$ psi.

6. Primary Loaded Beam

This experiment will provide a direct measure of the swelling-enhanced creep rate in bending. Specimens of high swelling 304 SS duct will be loaded in a device shown schematically in Figure HEDL-8. The bellows will maintain a nearly constant bending moment on the beams. The measured quantities are the bowing strain after irradiation, and the applied bending moment. The swelling-enhanced creep coefficient is given by:

$$B' = \frac{\epsilon}{\sigma t} = \frac{I}{MtR} \quad (13)$$

where ϵ is the maximum fiber strain, R is the specimen radius of curvature, I is the moment of inertia of the beam, and t is the irradiation time. The error in B' will be mainly due to uncertainties in the radius of curvature and changes in the bending momentum so that:

$$\left| \frac{\Delta B'}{B'} \right| = \left| \frac{\Delta R}{R} \right| + \left| \frac{\Delta M}{M} \right| = \left| \frac{16R\Delta Y}{\ell^2} \right| + \left| \frac{\Delta M}{M} \right| \quad (14)$$

where ℓ is the sample length.

The change in the bending moment is a result of the bowing strain in the beam which alters the effective lever arm distance L . This reduction in bending moment varies along the beam

FIGURE HEDL-7. Swelling-Driven Creep.

L_0 = LEVER ARM LENGTH (UNSTRAINED)
 $L(X)$ = LEVER ARM LENGTH AT X (STRAINED)

FIGURE HEDL-8. Primary Loaded Beam.

and is a maximum in the center ($X = 0$, Figure HEDL-8) where the bowing deflection is largest. The results for the calculated bending moment as a function of X , the position on the beam, are shown in Figure HEDL-9 for a range of maximum fiber strains of up to 0.0075 in magnitude. At a strain of 0.0075, the bending moment is reduced at most to 90% of its value at zero strain. As an example of the total uncertainty in the creep coefficient from both bending moment changes and radius of curvature measurements, a relatively large total strain of $\epsilon = 5 \times 10^{-3}$ or $R = 4$ inches gives $\left| \frac{\Delta M}{M} \right|_{\max} = 6.5\%$ and $\left| \frac{\Delta R}{R} \right| = 1.5\%$ yielding $\left| \frac{\Delta B'}{B} \right| = 8.0\%$.

The major conclusion to be drawn from this analysis is that the specimen designs selected for the history effects experiment have adequate sensitivity to determine if prior irradiation history has a significant impact on irradiation creep processes.

Further work in this area will be directed towards finalization of specimen design and fabrication techniques for preparing specimens from previously irradiated specimens.

B. VISCOELASTIC ANALYSIS OF STRESS RELAXATION IN A SLIT TUBE

G. L. Wire/J. L. Straalsund

1. Objective

The purpose of this work is to derive the time dependence for stress-relaxation in a slit tube. This information is needed to establish the utility of the slit tube specimen for in-reactor stress-relaxation studies.

2. Accomplishments and Status

This report will detail the results of a viscoelastic analysis for stress relaxation in a slit tube. In an earlier report⁽¹⁾ it was shown that a slit tube which was of suitable dimensions for in-reactor deformation work would deform in a state of plane strain. The more familiar example of viscoelastic stress relaxation involves a state of plane stress and in this case relaxation occurs according to a single exponential law:

$$\sigma = \sigma_0 e^{-t/t_r} \quad (15)$$

where σ_0 is the stress at time $t = 0$, t_r is the relaxation time, and σ is the instantaneous stress. For example, stress relaxation in a narrow bent beam or in a typical tensile specimen of small transverse dimensions compared to its length would relax according to Equation (15). However, the slit tube will have a more complicated relaxation dependence because of the fact that it is in a state of plane strain.

Before investing the considerable effort required to obtain stress-relaxation measurements on the slit tube, the ability to derive useful creep information from this test must be established. A rigorous analysis of stress relaxation in a slit tube is given below and it is shown that, in fact, in-reactor creep rate information can be derived from slit-tube stress relaxation data without serious error.

3. Relaxation Analysis

The model chosen for the calculation was that of an isotropic viscous solid with the plastic flow properties characterized by the viscosity constant μ . This model is consistent with the major experimental observations made to date on 20% CW 316 SS:

FIGURE 9. Bending Moment Variation.

FIGURE HEDL-10. Stress Relaxation Plots.

- 1) The material appears to creep essentially in steady state, although there can be a short initial transient effect.
- 2) The strain-rate appears to be linearly dependent upon applied stress.

It should be pointed out that the utility of the slit tube for in-reactor creep doesn't solely depend on the validity of the above listed observations; in fact, experiments on the slit tube are expected to provide a sensitive means of verifying or modifying this proposed description.

On the basis of the assumed viscous flow law the plastic strain-rates are of the form:⁽⁴⁾

$$\dot{\epsilon}_{rp} = \frac{1}{2\mu} [\sigma_r - 1/3(\sigma_r + \sigma_\theta + \sigma_z)] \quad (16a)$$

$$\dot{\gamma}_{r,\theta p} = \frac{\tau_{r\theta}}{\mu} \quad (16b)$$

In Equation (16) σ_r is the radial stress, σ_θ is the tangential stress and σ_z is the axial stress (see Figure HEDL-10) and the quantity $\dot{\epsilon}_{rp}$ is the radial strain rate, the symbol p signifying that it is the plastic component. The formulas for the tangential and axial strain-rates are easily obtained from permuting the coordinate indices in Equation (16) and are therefore not listed separately. The elastic behavior for the model chosen is described by:

$$\epsilon_{re} = \frac{1}{2G} [\sigma_r - \frac{\nu}{1+\nu} (\sigma_r + \sigma_\theta + \sigma_z)] \quad (17a)$$

$$\gamma_{r,\theta e} = \frac{\tau_{r\theta}}{G} \quad (17b)$$

where ν is the Poisson's ratio and G is the shear modulus. The solution we are seeking to describe stress-relaxation will be based on Equations (16) and (17). Further, the proposed solution must meet the following requirements:

- 1) The compatibility equation is satisfied.
- 2) The equilibrium conditions are satisfied.
- 3) The boundary conditions appropriate to stress relaxation are met.

These, of course, are the same requirements as are necessary in the purely elastic problem except for one difference. That is, the strain will now contain a plastic component, as well as an elastic component, as well as an elastic component.

$$\epsilon = \epsilon_p + \epsilon_e \quad (18)$$

and it is this total strain which must obey the compatibility equation.

The compatibility equation in polar coordinates for a two-dimensional problem is:

$$\frac{1}{r} \frac{\partial^2 \epsilon}{\partial \theta^2} + \frac{\partial}{\partial r} \left[\frac{\partial}{\partial r} (r \epsilon_\theta) - \epsilon_r \right] = \frac{1}{r} \frac{\partial^2 (r \gamma_{r\theta})}{\partial r \partial \theta} \quad (19)$$

and the two equilibrium equations are:

$$\frac{\partial}{\partial r} (r \sigma_r) - \sigma_\theta + \frac{\partial \tau_{r\theta}}{\partial \theta} = 0 \quad (20a)$$

$$\frac{\partial \sigma_{\theta}}{\partial \theta} + \frac{\partial}{\partial r} (r \tau_{r\theta}) + \tau_{r\theta} = 0 \quad (20b)$$

It is more useful to express the compatibility relation in terms of strain-rates rather than strains by differentiating Equation (19) with respect to time:

$$\frac{1}{r} \frac{\partial^2 \dot{e}_r}{\partial \theta^2} + \frac{\partial}{\partial r} \left[\frac{\partial}{\partial r} (r \dot{e}_{\theta} - \dot{e}_r) \right] = \frac{1}{r} \frac{\partial^2 (r \dot{\gamma}_{r\theta})}{\partial r \partial \theta} \quad (21)$$

The strain rates in Equation (21) are total strain rates and are given in terms of the stress as:

$$\begin{aligned} \dot{e}_r &= \dot{e}_{rp} + \dot{e}_{re} \\ &= \frac{1}{2\mu} [\sigma_r - 1/3 (\sigma_r + \sigma_{\theta} + \sigma_z)] \end{aligned} \quad (22a)$$

$$\begin{aligned} &+ \frac{1}{2G} \left[\dot{\sigma}_r - \frac{\nu}{1+\nu} (\dot{\sigma}_r + \dot{\sigma}_{\theta} + \dot{\sigma}_z) \right] \\ \dot{\gamma}_{r\theta} &= \frac{\tau_{r\theta}}{\mu} + \frac{\tau_{r\theta}}{G} \end{aligned} \quad (22b)$$

The problem at hand then is to calculate the stress distribution which satisfies Equations (20), (21) and (22) and is consistent with the boundary conditions for the slit tube. In the case of purely elastic flow, this procedure was fairly detailed and lengthy.⁽¹⁾ Fortunately, it is not necessary to go through a similarly involved procedure to derive a new stress distribution which satisfies Equations (20) and (21) and includes the effects of plastic flow. Rather, it is possible to generalize the elastic solution to describe plastic flow. The stress distributions for the elastic sen⁽¹⁾ can be written symbolically:

$$\sigma_r = P f_1 (r, \theta) \quad (23a)$$

$$\sigma_{\theta} = P f_2 (r, \theta) \quad (23b)$$

$$\sigma_z = \nu (\sigma_r + \sigma_{\theta}) = P (f_1 (r, \theta) + f_2 (r, \theta)) \quad (23c)$$

$$\tau_{r\theta} = P f_3 (r, \theta) \quad (23d)$$

where P is the applied load and f_1 , f_2 and f_3 are somewhat detailed functions of r and θ . As a guess in generalizing Equation (23) to include plastic flow and stress-relaxation, one might expect that the radial and angular dependence of the stress distribution is unchanged and that the only change is that the load P becomes time dependent, relaxing ultimately to zero as plastic strain commences. However, this postulate applied to Equation (23) leads to the stress σ_z still obeying the elastic plane strain condition, i.e., $\sigma_z = \nu (\sigma_r + \sigma_{\theta})$ or $\epsilon_{ze} = 0$. The appropriate plane-strain boundary condition now is $\dot{e}_z = 0$; that is that the total strain rate is zero along the length of the tube. Hence, a further generalization of Equation (23) is required. A satisfactory generalization is to take both P and ν to be time dependent so that the trial stress distribution is:

$$\sigma_r = P(t) [f_1(r, \theta)] \quad (24a)$$

$$\sigma_\theta = P(t) [f_2(r, \theta)] \quad (24b)$$

$$\sigma_z = \beta(t) [f_1(r, \theta) + f_2(r, \theta)] \quad (24c)$$

$$\tau_{r\theta} = P(t) f_3(r, \theta) \quad (24d)$$

where $\beta(t)$ can be viewed as the product of $P(t)$ and a time dependent Poisson's ratio.

It is easily seen that the stress distribution in Equation (24) satisfies the equilibrium condition (Equation 20) as the only changes are that the load P and Poisson's ratio ν are now time-dependent, but the continuity equation holds for any values of the load and Poisson's ratio. A straight-forward but tedious check shows that the compatibility equation is also satisfied by this form. This is done by using Equation (22) to calculate the strain-rates and substituting into Equation (21).

The task remaining is to calculate the functions $P(t)$ and $\beta(t)$ based on Equation (16) and the boundary conditions. The plane strain condition required $\dot{\epsilon}_z = 0$ for all times during the deformation, which leads to:

$$0 = \frac{1}{2\mu} [\sigma_z - 1/3 (\sigma_r + \sigma_\theta + \sigma_z)] + \frac{1}{2G} [\sigma_z - \frac{\nu}{1+\nu} (\dot{\sigma}_r + \dot{\sigma}_\theta + \dot{\sigma}_z)] \quad (25a)$$

which can be written as:

$$\dot{\sigma}_z + \frac{\sigma_z}{t_r} = \nu(\dot{\sigma}_r + \dot{\sigma}_\theta) + \frac{(\sigma_r + \sigma_\theta)}{2t_r} \quad (25b)$$

$$\text{where } t_r = \frac{3\mu}{2G(1+\nu)} = \frac{3\mu}{E}.$$

Using Equation (25b) the strain rates can be written as:

$$\dot{\sigma}_r = \frac{1}{2G} (\dot{\sigma}_r - \dot{\sigma}_z + \frac{\sigma_r - \sigma_z}{t_s}) \quad (26a)$$

$$\dot{\sigma}_\theta = \frac{1}{2G} (\dot{\sigma}_\theta - \dot{\sigma}_z + \frac{\sigma_\theta - \sigma_z}{t_s}) \quad (26b)$$

$$\dot{\gamma}_{r\theta} = \frac{\tau_{r\theta}}{Gt_s} \quad (26c)$$

$$\text{where } t_s = \frac{\mu}{G} = \frac{2(1+\nu)t_r}{3}$$

Substituting Equation (24) into Equation (25) leads to the following relationship between P and β :

$$\dot{\beta} + \frac{\beta}{t_r} = \dot{P}\nu + \frac{P}{2t_r} \quad (27)$$

Equation (27) is not sufficient to determine the functions $P(t)$ and $\beta(t)$, and to do this, another relationship is needed. This condition comes from the constraint that the slit width

V is constant during stress relaxation, i.e.:

$$\dot{V}_{O=0} = 0 \quad (27a)$$

where V is the total displacement in the O direction. The expression for $\dot{V}$ can be obtained in the same way as the displacement was not calculated from the elastic previously.⁽¹⁾ It is not necessary to explicitly do this step as it follows the same form as the calculation done in Reference 1 and the results are:

$$\dot{V}_{O=0} = \text{Const.} \times \left[\frac{(\dot{P} - \dot{\beta} \nu)}{G(1+\nu)} + \frac{1}{\mu} \left(\frac{2}{3} P - \frac{\beta}{3} \right) \right] = 0 \quad (28)$$

which can be rewritten:

$$\dot{P} - \nu \dot{\beta} = \frac{-(P - \beta/2)}{t_r} \quad (29)$$

It should be pointed out that Equation (28) involves a simplification in that the compliance of the wedging device was neglected. This approximation introduces only a negligible error as the compliance of the wedge, which will be thin (approximately 0.050 inch thick) is calculated to be less than 1% of that of the slit tube.

The coefficients P(t) and $\beta(t)$ are now determined by the two simultaneous linear differential equations, Equations (27) and (29). These can be solved by elementary means to yield:

$$P(t) = P_0 \left[\frac{(1+\nu)}{2} e^{-\frac{t}{2t_r(1-\nu)}} + \frac{(1-\nu)}{2} e^{-\frac{3t}{2(1+\nu)t_r}} \right] \quad (30a)$$

$$\beta(t) = P_0 \left[\frac{(1+\nu)}{2} e^{-\frac{t}{2t_r(1-\nu)}} + \frac{(1-\nu)}{2} e^{-\frac{3t}{2(1+\nu)t_r}} \right] \quad (30b)$$

Equation (30) is the final result of this calculation as it describes how the load P, which is the measurable quantity, relaxes with time due to the assumed viscous creep law. It shows that the slit tube relaxes with two different relaxation times, $2t_r(1-\nu)$ and $\frac{2(1+\nu)t_r}{3}$. For an incompressible solid ($\nu = 1/2$) there is of course only one relaxation time, which is just t_r . It is interesting to compare this result with that for a uniaxial specimen. If a uniaxial specimen relaxes under the normal plane stress conditions and the compliance of the fixture is ignored, the stress will decay with a relaxation $\sigma = \sigma_0 e^{-t/t_r}$. On the other hand, if the specimen is constrained to relax in a plane strain configuration, the stress will decay as:

$$\sigma = \sigma_0 \left[\frac{(1+\nu)}{2} e^{-\frac{t}{2t_r(1-\nu)}} + \frac{(1-\nu)}{2} e^{-\frac{3t}{2t_r(1+\nu)}} \right]$$

which is exactly the same as the expression in Equation (30). The fact that there are two relaxation times involved in the slit tube relaxation is just due to the fact that it is in plane strain. Essentially, the requirement that $\dot{\epsilon}_z = 0$ in plane strain leads to a complex time dependence for $\sigma_z(t)$ [or $\beta(t)$].

The intended use of the slit tube stress relaxation experiment is to provide a measure of the creep constant for the material. According to the viscoelastic model used in this work, a uniaxial specimen would creep at a rate $\dot{\epsilon} = \frac{\sigma}{3\mu}$ so that creep constant $(\dot{\epsilon}/\sigma)$ is given as $\frac{1}{t_r E}$. The ability to infer a creep coefficient from slit tube stress relaxation data is then determined by the ability to determine the quantity t_r from the data. Several approaches are possible in this regard. One approach is to make a computer fit of Equation (30) to the stress-relaxation data and generate a best fit value of t_r . To justify this approach, fairly detailed data would be required and this would probably not prove to be feasible with data from in-reactor creep. A second approach is to approximate Equation (30) over the range of interest to be a single relaxation time. Figure HEDL-10 is a plot of Equation (30) over relatively wide experimental range of load P for a typical value of $\nu = 0.3$. The function in Equation (30) is quite close to a straight line, in fact for $0.2 \leq P/P_0 < 1$ the function fits the expression $e^{-t/1.20 t_r}$ to within a 2% error. Thus, the major change that the extra term due to plane strain relaxation introduces (for a reasonable range of load drop and for a typical value of ν) can be described as an increase in the relaxation time by about 20% over that occurring in plane stress.

Using this observation, it should be a simpler matter to derive the creep coefficient from the stress relaxation without loss of accuracy.

4. Conclusions And/Or Recommendation

Results of the present investigation lead to the overall conclusion that data from in-reactor stress-relaxation of slit tubes promises to be a useful quantitative tool in determining creep coefficients of structural materials. In addition, the fact that plane-strain configuration in the slit tube specimen leads to two relaxation times has significance for other experiments as well. For example, in beam bending experiments, the beam width cannot be made small compared to its thickness without elaborate experimental design, so that plane strain conditions will prevail there as well. These experiments must therefore be analyzed according to the formulation developed in this work in order to accurately assess the creep constants from the data.

5. References

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3. L. C. Walters, Monthly Development Program Progress Report, ANL-RRD-20, September 1973, p. 113.
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C. EFFECT OF SIMULATED WEAR ON THE MECHANICAL PROPERTIES OF UNIRRADIATED 20% CW TYPE 316 STAINLESS STEEL PROTOTYPIC CLADDING

G. D. Johnson, C. W. Hunter/J. L. Straalsund, J. J. Holmes

1. Objective

The purpose of this work is to determine the effect of simulated wear marks on the deformation and failure behavior of 20% CW 316 SS prototypic FTR cladding. This information will

provide a basis for incorporating the effect of wear into the design and performance analysis of fast reactor fuel pins.

2. Scope of Work

Performance analysis of fast reactor fuel elements requires a quantitative description of the effects of cladding wastage on the mechanical properties of the cladding. One possible type of wastage is a wear mark on the outer surface of the cladding resulting from interaction with the wire wrap. Simulated wear marks were produced on the surface of 20% CW 316 SS tubing samples. Both stress-rupture and transient heating (FCTT) tests were conducted on these samples. Test results have been analyzed with respect to net section stress and Larson-Miller Parameter.

3. Test Material

The 20% CW 316 SS tubing used for the tests was unirradiated N-lot material. The wear marks were introduced by filing a flat spot on the outside surface of the specimen. The wear marks were either 0.25 or 1.0 inch in length and the depths studied were 0.001, 0.003 and 0.005 inch. The specimens were 2.5 inches long, with a nominal diameter and wall thickness of 0.230 and 0.015 inch.

4. Results

Both transient heating and stress rupture tests were performed to evaluate the effects of the simulated wear marks. Transient tests similar to those described in Refs. 1 through 3 were conducted at heating rates of 10°F/sec and 200°F/sec with an internal gas pressure of 10,000 psi. These conditions produced failures from 1150 to 1400°F. Biaxial stress rupture tests were conducted at 1000°F and 1300°F. Twelve control stress rupture tests were also conducted at 1300°F to establish a reference for comparison. Control tests were not performed at 1000°F, since previous testing results at 1000°F on N-lot material was available.⁽⁴⁾ Results of the transient test are summarized in Table HEDL-1 and the stress rupture tests are listed in Table HEDL-2.

It is necessary to describe in a quantitative manner the effects on mechanical properties produced by a wear mark. Analysis of the data from Table HEDL-2 has shown that the use of a net-section stress adequately describes the effect of wear marks. The net-section stress is computed assuming the wall thickness has been uniformly reduced by an amount equal to the depth of the wear mark. The validity of this approach is shown in Figure HEDL-11, where the net-section hoop stress is given as a function of time to rupture at 1000 and 1300°F. Control data for 1000°F was obtained from Ref. 4. From this figure, it is obvious that the use of net-section stress describes the effect of the wear mark within the normal scatter of the biaxial stress rupture control data.

The results of the transient tests are consistent with the stress rupture data. A failure correlation⁽⁵⁾ has been developed for tensile, burst, stress-rupture and transient tests of 20% CW 316 SS tubing. This failure correlation uses the life fraction rule in connection with the Larson-Miller Parameter. This approach was used to evaluate the wear mark data of Tables HEDL-1 and 2. The net-section stress for specimens with wear marks are plotted in Figure HEDL-12 with the basic correlation curve from Ref. 5. The specimens with wear marks essentially lie on the basic failure correlation curve. This agreement further demonstrates the validity of using the net-section stress approach for calculating the effect of wear marks on load-bearing capability.

TABLE HEDL-1
TRANSIENT TEST RESULTS

Specimen Number	Heating Rate, F°/sec	Wear Mark Dimensions		Failure Temp., °F	Nominal Hoop Stress, ksi	Net Section Hoop Stress, ksi
		Depth, in.	Length, in.			
W4	200	0.003	1.0	1145	72.0	90.0
W15	200	0.001	1.0	1370	72.0	77.1
W16	200	0.003	0.25	1310	72.0	90.0
W3	10	0.003	1.0	1240	72.0	90.0
W8	10	0.001	1.0	1290	72.0	77.1
W10	10	0.003	0.25	1255	72.0	90.0
W13	10	0.001	0.25	1315	72.0	77.1

TABLE HEDL-2
STRESS RUPTURE RESULTS

Specimen Number	Temp., °F	Wear Mark Dimensions		Rupture Time, hr.	Nominal Hoop Stress, ksi	Net Section Hoop Stress, ksi
		Depth, in.	Length, in.			
WC1	1300	0	0	1109.6	16.0	16.0
WC3				125.3	25.0	25.0
WC4				403.4	21.0	21.0
WC5				685.4	17.0	17.0
WC6				2956.5	14.0	14.0
WC7				4491.1	10.0	10.0
WC8				3972.3	11.0	11.0
WC9				3009.0	12.0	12.0
WC10				2314.2	13.0	13.0
WC11				1477.6	15.0	15.0
WC13				743.0	18.0	18.0
WC14				481.5	20.0	20.0
W19	1300	0.005	1.0	155.6	16.37	24.56
W20		0.005	1.0	265 ± 36	13.4	20.1
W23		0.003	1.0	457.8	16.37	20.46
W24		0.003	1.0	634.6	14.81	18.51
W25		0.003	1.0	1289 ± 19.8	13.4	16.75
W26		0.001	1.0	883.9	16.37	17.53
W27		0.001	1.0	1794.8	14.81	15.86
W17	1000	0.005	1.0	178.0	57.0	85.5
W18		0.005	1.0	330.1	46.98	70.47
W21		0.003	1.0	594.3	51.76	64.7
W22		0.003	1.0	985.1	46.98	58.73
W2		0.003	1.0	288.0	57.0	71.25
W9		0.003	0.25	439.0	57.0	71.25
W7		0.001	1.0	984.0	57.0	61.05
W12		0.001	0.25	1555.0	57.0	61.05

FIGURE HEDL-11. Stress Rupture Properties of 20% CW 316 SS Tubing with Simulated Wear Mark.

FIGURE HEDL-12. Effect of Simulated Wear Marks on the Mechanical Properties of 20% CW 316 SS.

6. References

1. R. L. Fish, C. W. Hunter and J. J. Holmes, "Strain Response and Failure of 20% Cold Worked Type 316 Stainless Steel Cladding During Temperature Transients," HEDL Quarterly Technical Progress Report.
2. C. W. Hunter and R. L. Fish, "Deformation and Failure of Fast Reactor Fuel Cladding Specimens During High-Temperature Simulated Loss-Of-Flow Transients," ANS Fast Reactor Safety Conference, April 2-4, 1974, USAEC-Conf. 740401.
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D. LOW TEMPERATURE CREEP OF 20% CW 316 SS

L. D. Blackburn, R. W. Barker, T. R. Billeter/J. L. Straalsund

1. Objective

The objectives of this work are: (1) to accurately measure thermal creep in 20% CW 316 SS for comparison with in-reactor creep results; and (2) to further demonstrate the application of microwave extensometers for high precision creep strain measurements.

2. Scope of Work

A constant-load creep test was performed on 20% CW 316 SS at 850°F under a stress of 20,000 psi using a high precision microwave extensometer to measure strain. Details of specimen and extensometer design were described previously.⁽⁶⁾ The specimen was manufactured from the same heat of steel employed in an earlier in-reactor experiment,⁽⁷⁾ but the grain size in the present specimen is ASTM 6 or an average grain intercept of 40 microns, compared to a grain size of ASTM 7 (average grain intercept 25 microns) for the in-reactor test material.

The specimen was heated to 800°F in about 75 hours, was held at approximately 800°F for 175 hours, and then heated to 850°F and held for 15 hours prior to applying the test load. Temperature control during the test was within $\pm 0.5^\circ\text{F}$. The test environment was a gas mixture of 94% helium - 6% hydrogen, employed to prevent oxidation of the pure copper linings in the microwave sensors.

Each microwave sensor of the averaging extensometer was calibrated separately at room temperature using a precision micrometer. Calibration displacement was correlated with sensor frequency using a third degree polynomial in frequency. For one sensor, the standard error for the calibration displacement was 12.9 microinches and the maximum deviation was 25.4 microinches. For the other sensor, the standard error and maximum deviation were 19 and 37 microinches, respectively. The accuracy of the calibration micrometer was not better than 20 microinches. The measured specimen gage length at room temperature was 2.9997 inches, so the extensometer qualifies as ASTM Class A. All strains were computed based on the room temperature specimen gage length and sensor calibrations.

3. Results

The specimen was loaded in increments of about 2000 psi, and the deformation was totally elastic, as shown in Figure HEDL-13. The experimental value of Young's modulus was 24.5×10^6 psi, in good agreement with reported values for Type 316 SS.⁽⁸⁾

FIGURE HEDL-13. Elastic Loading of 20% CW 316 SS at 850°F.

HEDL-21

FIGURE HEDL-14. Creep Curve for 20% CW 316 SS at 850°F/20,000 psi.

Time-dependent creep deformation measurements are presented in Figure HEDL-14. These data were fit to a fourth degree polynomial in $t^{0.15}$, and the standard error of the estimate of creep strain was 1×10^{-6} . Essentially identical results were obtained with polynomials of degree five and six. The polynomial relation was differentiated to obtain creep rate and the results are shown in Figure HEDL-15. Creep rate decreases by almost three orders of magnitude over the 1000-hour test, and there is no indication of approach to a minimum or steady-state creep rate. Creep strains measured in an in-reactor (EBR-II) test⁽⁷⁾ under the same conditions were about seven times larger than strains reported here, as shown in Figure HEDL-16.

FIGURE HEDL-15. Creep Rate as a Function of Creep Strain in 20% CW 316 SS at 850°F/20,000 psi.

At 1031 hours, the specimen was unloaded to a stress of 220 psi. The measured elastic contraction agreed with the elastic loading strain to within 6×10^{-6} . Initial measurements indicate significant time-dependent strain recovery in the unloaded condition, and these results will be presented in future reports.

4. Evaluation of Results

Several factors affect the accuracy of the strain measurements reported here. First, of course, is the calibration procedure, which was described earlier. The microwave sensors

were calibrated at room temperature, but were then used at 850°F in the creep test. The potential error associated with this difference between calibration temperature and operation temperature was estimated from the theory relating microwave frequency, sensor dimensions and mode of excitation; it was found that the ratio of strain values based on room temperature calibration to strain values based on 850°F calibration should be about 1.006. Small changes in sensor radius were detected during the creep test by monitoring a mode frequency which depends only on sensor radius. However, these sensor radius changes in themselves affect the frequency of the mode, which is sensitive to length changes, and therefore introduce an additional error in reported strains. The measured radius changes and the associated correction to axial strain as computed from the theoretical relation between resonant frequency and sensor dimensions are shown in Figure HEDL-16a. These changes in sensor dimensions may result from reaction between hydrogen from the test atmosphere and dissolved oxygen in the OFHC copper liner of the sensor, but they are much smaller than the effects observed for impure copper liners.⁽⁶⁾

Several analytical representations were examined in formulating a quantitative description of the strain-time data. For the low temperature and stress and the short time of this test, steady-state creep was not expected to contribute significantly to the observed strains, and this was confirmed by the polynomial representation (see Figure HEDL-15). Therefore, the following analytical forms were considered in describing the creep strain (ϵ_c) data:

$$\epsilon_c = At^m \quad (1)$$

$$\epsilon_c = \epsilon_0 + At^m \quad (2)$$

$$\epsilon_c = A + B \ln t \quad (3)$$

$$\epsilon_c = B \ln (1 + t) \quad (4)$$

$$\epsilon_c = B \ln (1 + ct) \quad (5)$$

$$\epsilon_c = A (1 - e^{-Bt}) \quad (6)$$

$$\epsilon_c = t/(A + Bt) \quad (7)$$

$$\epsilon_c = t/(A + Bt^m) \quad (8)$$

None of these representations were completely satisfactory in describing the experimental data, and the polynomial representation described earlier was selected as the best description.

5. Conclusions And/Or Recommendations

Thermal creep strain in 20% CW 316 SS at 850°F and 20,000 psi is small, reaching a value of 1.2×10^{-4} in 1000 hours. Even at these low strain levels, the course of creep deformation can be followed in detail using microwave extensometry. Creep strain measured in the present experiment is substantially smaller than in-reactor creep strain determined under the same test conditions.

FIGURE HEDL-16. Comparison of Laboratory Creep Test with In-Reactor Creep Test.

FIGURE HEDL-16A. Variation of Microwave Sensor Radius During Creep Test and Associated Axial Strain Correction.

Results of stress changes (unloading and reloading) on creep behavior will be obtained during the next reporting period.

6. References

4. M. M. Paxton, "Mechanical Properties of Prototypic FTR Cladding--20% CW 316 Stainless Steel Tubing," HEDL-TME 71-59.
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6. L. D. Blackburn, R. W. Barker and T. R. Billeter, HEDL Quarterly Technical Progress Report - July, August, September 1973, HEDL-TME 73-5, Vol. 1, October 1973, p. B-1.

E. EFFECT OF NEUTRON FLUENCE ON THE MECHANICAL PROPERTIES OF 20% CW 316 SS

G. D. Johnson/J. L. Straalsund, J. J. Holmes

1. Objective

The objective of this work is to determine the effect of neutron fluence on the mechanical properties of 20% CW 316 SS prototypic FTR cladding. This information supports the design and performance analysis of fast reactor fuel pins.

2. Scope of Work

Recent work at HEDL describe the application of the life fraction rule and Larson-Miller Parameter (LMP) for correlation of transient test data with conventional mechanical properties data for 20% CW 316 SS prototypic FTR cladding, and this work is to be issued as a formal report. Use of this correlation procedure yielded a relationship between the modulus stress and the Larson-Miller Parameter. Data for tensile, stress-rupture, burst and transient tests were reduced to a single curve described by $\sigma = f(\text{LMP})$. This report describes the use of this correlation for analyzing the effect of neutron fluence ($E > 0.1$ MeV) on the mechanical properties of 20% CW 316 SS cladding.

3. Use of Life Fraction Rule and Larson-Miller Parameter

Data will be presented for the effect of neutron fluence on the tensile strength, stress-rupture life, and transient behavior for 20% CW 316 SS cladding. Before this is done, the method of calculating the LMP for each type of test will be summarized in the forthcoming report mentioned above.

The application of the Larson-Miller Parameter to the stress-rupture data is conducted in the standard manner, namely

$$\text{LMP} = T [\log t_r + 20] \quad (9)$$

where T = absolute temperature, $^{\circ}\text{R}$

t_r = time to rupture, hours.

For the tensile tests, the total elongation for each test is divided by the strain rate to provide a value for time to rupture in calculating the LMP. The ultimate tensile strength is used as the stress level for correlation with the LMP. Also, plastic stability considerations are used to make stress state corrections on the UTS data obtained in the athermal deformation region.

Larson-Miller Parameters for the thermal transient tests are determined by integrating, on a life fraction basis, the temperature-time path during the test to the point of failure. The following is the equation for the transient tests:

$$\frac{LMP}{T^*} + \log \left[\frac{LMP}{T^*} \right] = 16.08 - \log \left[\frac{\dot{T}}{T^*} \right] \quad (10)$$

where T^* = failure temperature, °R

$\dot{T}$ = heating rate, °F/sec.

As explained in the development of the correlation, all stresses are modified according to the stress parameter

$$\sigma = \sigma_T \left(\frac{E_{1400}}{E_T} \right) \quad (11)$$

where σ_T = stress at temperature T

E_{1400} = modulus at 1400°F

E_T = modulus at temperature T.

4. Experimental Data

Numerous post-irradiation mechanical property tests have been conducted on fuel pin cladding and cladding material. In all cases, the material has been 20% CW 316 SS. Fluences reported are $E > 0.1$ MeV.

Tensile tests⁽⁹⁾ have been conducted on cladding which has been irradiated to neutron fluences of 0.9×10^{22} n/cm². These tests were conducted in vacuum at an initial strain rate of 1.82×10^{-3} /min in the temperature range of 900 to 1600°F. These data, with the appropriate modulus modified stress and LMP values, are presented in Table HEDL-3.

Post-irradiation stress rupture behavior of cladding has been evaluated for fluences to 0.9×10^{22} n/cm² at test temperature of 1000, 1200 and 1400°F.⁽¹⁰⁾ These tests were performed by pressurizing tubular specimens to a predetermined level and maintaining a constant temperature and pressure until rupture. Table HEDL-4 presents the irradiation and test conditions with the respective LMP and modulus modified stress values.

A few stress rupture tests have been conducted on specimens from fuel pins and on a specimen from a thermocouple conduit tube.^(11,12) Table HEDL-5 summarizes the irradiation conditions, test parameters, stress values and Larson-Miller Parameter values.

Numerous transient tests have been conducted on irradiated cladding specimens and specimens from fuel pins for fluences up to 2.65×10^{22} n/cm².^(12,13) Failure temperatures ranged from 1020 to 2380°F. Tables HEDL-6 through 9 summarize the irradiation and test conditions and present the correlation values for stress and the LMP.

4. Results and Discussion

To facilitate interpreting the effect of fluence on the mechanical properties of the cladding, it has been necessary to group the data into three regions as: $< 1 \times 10^{22}$ n/cm², $> 1 \times 10^{22}$ n/cm² and $< 2.4 \times 10^{22}$ n/cm², and finally, $> 2.4 \times 10^{22}$ n/cm² and $< 3.5 \times 10^{22}$ n/cm². There are not enough data to permit a grouping of more regions of fluence. The three regions chosen are arbitrary and are based only on convenience. This grouping is reflected in the ordering of the transient data in Tables HEDL-6 through 9.

TABLE HEDL-3
POST-IRRADIATION TENSILE PROPERTIES OF 20% CW 316 SS CLADDING
Strain Rate = 1.82×10^{-3} /min

Specimen Number	Fluence, 10^{22} n/cm ² E > 0.1 MeV	Irrad. Temp., °F	Test Temp., °F	UTS, ksi	Equivalent Hoop Stress, ksi	Modulus Modified Stress, ksi	Total Elong., %	Test Time, hr.	Larson-Miller Parameter $\times 10^{-4}$
G2	0.5	865	900	94.4	104.8	85.2	9.0	0.824	2.71
G3	0.5	865	1100	85.2	94.6	83.2	5.4	0.495	3.07
G4	0.5	865	1200	60.9	60.9	56.3	8.5	0.778	3.30
G5	0.5	865	1400	35.5	35.5	35.5	11.0	1.007	3.72
G6	0.5	865	1600	17.7	17.7	19.3	6.4	0.566	4.07
BB2	0.2	960	1000	91.1	101.1	85.4	7.2	0.659	2.89
BB3	0.2	960	1200	64.0	64.0	59.2	5.4	0.495	3.27
BB4	0.2	960	1400	37.0	37.0	37.0	5.5	0.504	3.66
BB5	0.2	960	1600	19.3	19.3	21.1	4.4	0.403	4.04
F2	0.75	1040	1000	79.5	84.2	71.1	7.5	0.687	2.90
F3	0.75	1040	1200	61.7	61.7	57.1	7.0	0.641	3.29
F4	0.75	1040	1400	36.4	36.4	36.4	1.4	0.128	3.55
F5	0.75	1040	1600	15.8	15.8	17.3	1.3	0.119	3.93
C17	0.75	1110	1000	76.7	85.1	71.9	9.4	0.861	2.91
E2	0.9	1440	1400	26.6	26.6	26.6	2.2	0.201	3.59
F3	0.9	1440	1600	12.6	12.6	13.8	1.8	0.165	3.96
D2	0.9	1550	1500	14.0	14.0	14.6	3.3	0.302	3.82
D3	0.9	1550	1600	9.9	9.9	10.8	2.5	0.229	3.99

TABLE HEDL-4
POST-IRRADIATION BIAXIALY LOADED STRESS-RUPTURE TESTS ON 20% CW 316 SS

Specimen No.	Fluence, 10^{22} n/cm ² E > 0.1 MeV	Irrad. Temp., °F	Test Temp., °F	Hoop Stress (psi)	Modulus Modified Stress (ksi)	Rupture Life, hrs.	Larson-Miller Parameter $\times 10^{-4}$
BB-42	0.207	950	1000	65,000	55.2	137	3.22
EB-43	0.207	950	1000	60,000	51.0	200	3.26
BB-44	0.207	950	1000	55,000	46.7	574	3.22
C-42	0.738	1060	1000	65,000	55.2	191	3.25
C-43	0.738	1050	1000	60,000	51.0	446	3.30
C-44	0.738	1050	1000	55,000	46.7	1616	3.40
F-42	0.738	975	1000	65,000	55.2	66	3.19
F-44	0.738	970	1000	54,600	46.7	243	3.26
F-43	0.738	970	1000	55,000	46.7	79	3.20
F-46	0.738	970	1000	50,000	42.5	215	3.26
BB-49	0.207	950	1200	34,000	31.4	345	3.74
BB-50	0.207	950	1200	29,000	26.8	1776	3.86
C-50	0.738	1050	1200	29,000	26.8	955	3.82
E-42	0.900	1330	1400	17,000	17.0	31	4.00
E-43	0.900	1320	1400	12,000	12.0	111	4.10
E-44	0.900	1320	1400	8,000	8.0	835	4.26
D-44	0.900	1440	1400	8,000	8.0	108	4.10
D-42	0.900	1450	1400	7,000	7.0	73	4.07

TABLE HEDL-5
POST-IRRADIATION BIAXIALLY-LOADED STRESS-RUPTURE TESTS
on 20% CW 316 SS CLAD FROM FUEL PINS

Specimen Number	Fluence (10^{22} n/cm ²) E>0.1 MeV	Irrad. Temp. (°F)	Test Temp. (°F)	Hoop Stress (ksi)	Modulus Modified Stress (ksi)	Rupture Life (hr)	Larson-Miller Parameter x 10 ⁻⁴
PNL-17-GH	2.0	1020	1200	27.5	25.4	417	3.75
PNL-17- Conduit G3	2.0	1020	1200	27.5	25.4	657	3.88
P23A:58B-M	0.64	1130	1200	34.0	31.5	80	3.64
P23A:58B-O	0.56	1260	1300	22.2	21.3	68	3.84
P23A:58B-S	0.40	1300	1300	22.2	21.3	71	3.85
P23A:58B-Y	0.06	1260	1300	22.2	21.3	63.4	3.84
N-070:O	1.3	1105	1000	57.0	45.6	319	3.28
N-070:S	0.11	1100	1000	57.0	45.6	403	3.30

TABLE HEDL-6
THERMAL TRANSIENT TEST DATA
(Fluence < 1×10^{22} n/cm², E > 0.1 MeV)

Specimen Number	Fluence (10^{22} n/cm ²) E>0.1 MeV	Irrad. Temp. (°F)	Hoop Stress (ksi)	Modulus Modified Stress (ksi)	Thermal Ramp Rate (°F/sec)	Fail Temp. (°F)	Larson-Miller Parameter x 10 ⁻⁴
E12	0.9	1410	6.48	8.95	10	2060	4.33
E14	0.9	1410	6.48	9.65	200	2210	4.24
E11	0.9	1420	3.6	5.35	10	2200	4.58
E13	0.9	1420	3.6	5.55	200	2280	4.36
C52	0.74	1050	5.4	7.35	10	2030	4.28
C46	0.74	1050	5.4	7.90	200	2170	4.18
C51	0.74	1060	3.24	4.79	10	2190	4.57
C47	0.74	1050	3.24	4.83	200	2210	4.24
E16	0.9	1410	1.3	2.10	10	2380	4.91

TABLE HEDL-7
THERMAL TRANSIENT TEST DATA
(Fluence < 1×10^{22} n/cm², E > 0.1 MeV)

Specimen Number	Fluence (10^{22} n/cm ²) E > 0.1 MeV	Irrad. Temp., °F	Hoop Stress (ksi)	Modulus Modified Stress (ksi)	Thermal Ramp Rate (°F/sec)	Fail Temp. (°F)	Larson-Miller Parameter x 10 ⁻⁴
N-035K	0.7	1100	43.2	45.2	10	1505	3.34
N-035J	0.95	1105	43.2	46.0	10	1540	3.41
P23A-28R-0	0.55	1170	43.2	45.5	10	1520	3.37
N-005K	0.7	1070	28.8	33.4	200	1705	3.44
N-035N	0.3	1080	28.8	35.0	200	1820	3.60
N-035M	0.3	1080	48.2	47.3	200	1600	3.27

TABLE HEDL-8
THERMAL TRANSIENT TEST DATA
(Fluence > 1×10^{22} n/cm² and < 2.4×10^{22} n/cm², E > 0.1 MeV)

Specimen Number	Fluence (10^{22} n/cm ²) E > 0.1 MeV	Irrad. Temp., °F	Hoop Stress (ksi)	Modulus Modified Stress (ksi)	Thermal Ramp Rate (°F/sec)	Fail Temp. (°F)	Larson-Miller Parameter x 10 ⁻⁴
N-035I	1.2	1105	28.8	32.0	10	1630	3.57
N-159D	2.2	835	72.0	61.9	10	1020	2.52
N-005B	1.9	765	97.2	84.2	10	1040	2.75
N-027I	1.45	1095	18.0	23.1	200	1920	3.76
N-005J	1.1	1075	28.8	33.5	200	1710	3.45
N-159C	1.9	770	43.2	45.3	200	1520	3.13
N-005H	1.65	1070	43.2	45.0	200	1490	3.09
N-159M2	1.2	1105	43.2	49.4	200	1680	3.40
N-070D	2.15	778	72.0	66.7	200	1210	2.63
N-027C	1.9	768	97.2	87.0	200	1120	2.48

TABLE HEDL-9
THERMAL TRANSIENT TEST DATA
(Fluence > 2.4×10^{22} n/cm² and < 3.5×10^{22} n/cm², E > 0.1 MeV)

Specimen Number	Fluence (10^{22} n/cm ²) E > 0.1 MeV	Irrad. Temp., °F	Hoop Stress (ksi)	Modulus Modified Stress (ksi)	Thermal Ramp Rate (°F/sec)	Fail Temp. (°F)	Larson-Miller Parameter x 10 ⁻⁴
N-005D	2.65	893	28.8	27.8	10	1325	3.03
N-027D	2.6	830	43.2	38.0	10	1130	2.71
N-035C	2.6	835	43.2	39.4	10	1170	2.75
N-035D	2.65	905	43.2	38.8	10	1125	2.70
N-027F	2.65	968	72.0	66.0	10	1175	2.76
N-005E	2.65	958	18.0	23.3	200	1940	3.80
N-005C	2.6	835	28.8	32.6	200	1670	3.39
N-035E	2.65	975	28.8	30.4	200	1525	3.15
N-C27E	2.65	900	72.0	69.4	200	1310	2.79

Data from Tables HEDL-3 through 9 were plotted as $\sigma = f(\text{LMP})$ for the three ranges of fluence in Figures HEDL-17 through 19. The curves through the data points represent a polynomial fit of the data. Equations for each fluence range and the range of applicability are:

$\phi t < 1 \times 10^{22} \text{ n/cm}^2$:

$$\text{LMP} = 4.61099 - 4.53547 \times 10^{-2} \sigma + 5.43856 \times 10^{-4} \sigma^2 - 3.04228 \times 10^{-6} \sigma^3$$

for $\sigma = 4$ to 94 ksi

$\phi t > 1 \times 10^{22} \text{ n/cm}^2$ and $< 2.4 \times 10^{22} \text{ n/cm}^2$:

$$\text{LMP} = 5.14967 - 0.06371 \sigma + 4.01133 \times 10^{-4} \sigma^2$$

for $\sigma = 20$ to 80 ksi

$\phi t > 2.4 \times 10^{22} \text{ n/cm}^2$ and $< 3.5 \times 10^{22} \text{ n/cm}^2$:

$$\text{LMP} = 4.81194 - 0.05652 \sigma + 9.49638 \times 10^{-5} \sigma^2$$

for $\sigma = 20$ to 45 ksi.

The equations for each fluence region are plotted with the curve for the unirradiated cladding correlation in Figure HEDL-20. For fluences $< 1 \times 10^{22} \text{ n/cm}^2$, there is a small reduction in stress for a given LMP for most of the curve. At low LMP's (athermal flow region) the irradiated cladding is stronger than the unirradiated cladding for low fluence. The curves for the other two fluence regions exhibit a sharp reduction in strength for a given LMP value. For the high fluence region, this amounts to about a factor of two in reduction of stress at the low LMP values. It appears that there is not as large an effect at higher LMP values for the higher fluences, although it should be noted that there are not enough data at higher LMP values to allow a prediction of the trend of curves B and C.

There is significant scatter in the data in Figures HEDL-18 and 19. At the present, considerable error could result in the prediction of behavior at the higher fluences if the equations for these fluence regions are used. The equations are presented only to show the trend of the effect of fluence on the LMP correlation.

This correlation presented in Figures HEDL-17 to 19 ignores any effects that may be due to irradiation temperature and time at the irradiation temperature. Future results from transient tests conducted on cladding from higher temperature fuel pins may provide enough data to permit an evaluation of the effect of irradiation temperature. Any effects due to time at temperature cannot be evaluated presently due to lack of data.

Also, the correlation does not analyze the effect of any fuel-cladding interaction. Metallographic analysis is currently being conducted and results from this effort may yield information which could affect the trend of the correlation.

5. Conclusions

It has been shown that a correlation developed for unirradiated cladding can be used to provide a good correlation for irradiated cladding. The correlation can be used to show the effect of neutron fluence on the mechanical properties of 20% CW 316 SS. Future work needs to determine the effect of irradiation temperature and time at temperature on the correlation.

Figure HEDL-17. Correlation of Mechanical Properties Data for Neutron Fluences of $<1 \times 10^{22} \text{ n/cm}^2$.

Figure HEDL-18. Correlation of Mechanical Properties Data for Neutron Fluences of $>1 \times 10^{22}$ to $<2.4 \times 10^{22} \text{ n/cm}^2$.

Figure HEDL-19. Correlation of Mechanical Properties Data for Neutron Fluences of $>2.4 \times 10^{22}$ and $<3.5 \times 10^{22}$ n/cm².

Figure HEDL-20. Effect of Neutron Fluence on the Mechanical Properties of 20% C.W. Type 316 Stainless Steel.

6. References

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9. R. L. Fish, "Postirradiation Tensile Properties of 20% CW Type 316 Stainless Steel," Monthly Technical Progress Report, August 1972, HEDL-TME 72-8.
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II. MATERIALS PERFORMANCE AND SURVEILLANCE

T. T. Claudson

A. HIGH STRAIN RATE EFFECTS

(Last Reported October-November-December 1973)

J. M. Steichen/R. L. Knecht, J. J. Holmes

1. Accomplishments and Status

High strain rate testing of thermally aged 304 SS has been completed. Specimens were aged in static sodium at temperatures of 800, 1000 and 1200°F for exposure times of 1000, 3500 and 10,000 hours. Testing was performed at the aging temperatures and strain rates from 3×10^{-5} to 10 sec^{-1} . Data from these tests included ultimate and 0.2% yield strengths, total and uniform elongations, and reduction of area. These data are listed in Table HEDL-10 and illustrated in Figure HEDL-21.

The effect of strain rate on the ultimate and 0.2% yield stress of thermally aged 304 SS for aging and test temperatures of 800, 1000 and 1200°F is presented in Figures HEDL-21 through 23, respectively. Included in the figures are data representing unaged behavior. No effect of thermal aging on strength properties is observed.

At aging and test temperatures of 800 and 1000°F, the ductility (total and uniform elongation) of the aged and unaged materials is essentially identical for all strain rates (Figures HEDL-24 and 25). Ductility at 1200°F is slightly influenced by thermal aging as shown in Figure HEDL-26. At the lowest strain rate, the total elongation of the aged material is significantly higher than the unaged material. As strain rate increases, the aged material total elongation is reduced and at a rate of 10 in./in./sec, it is slightly lower than the unaged material. The uniform elongation of the aged material at 1200°F is essentially identical to that of the unaged material to a rate of $\sim 0.01 \text{ in./in./sec}$ and is slightly lower for higher strain rates.

2. Expected Achievements

A topical report describing the influence of thermal aging on the high strain rate tensile properties of 304 SS will be prepared, and the effect of strain rate on the tensile properties of A-286, the IVHM bolting material, will be completed.

3. References

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TABLE HEDL-10
TENSILE PROPERTIES OF THERMALLY AGED 304 SS

Aging Conditions		Test Condition		Strength, (ksi)		Elongation (%)		Reduction of Area, (%)
Temp (°F)	Exposure (hr)	Temp (°F)	Strain Rate (sec ⁻¹)	0.2% YS	Ult	Total	Uniform	
800	1000	800	3 x 10 ⁻⁵	15.6	65.6	41.5	33.7	65.6
			3.5 x 10 ⁻³	16.4	61.4	36.7	30.9	64.8
			0.1	16.7	58.9	36.4	31.2	69.6
			1.0	16.0	56.2	36.0	31.4	72.0
			10.0	21.3	59.3	36.0	30.4	74.0
800	3500	800	3 x 10 ⁻⁵	14.2	64.7	41.4	37.6	65.0
			3 x 10 ⁻⁵	14.3	65.3	36.3	31.8	63.4
			3.5 x 10 ⁻³	16.3	60.7	37.0	32.2	65.0
			0.1	21.7	56.6	37.0	32.8	56.1
			10.0	20.0	57.4	34.9	29.2	68.8
800	10,000	72	3 x 10 ⁻⁵	33.1	84.6	74.8	66.7	79.7
		800	3 x 10 ⁻⁵	15.9	63.0	39.8	36.1	67.2
		800	3.5 x 10 ⁻³	16.7	62.3	37.2	32.8	72.4
			1.0	16.5	56.3	35.8	30.6	77.8
1000	1000	1000	3 x 10 ⁻⁵	15.4	56.6	37.0	31.6	64.6
			3.5 x 10 ⁻³	18.7	50.5	36.5	30.7	71.5
			0.1	15.0	54.1	34.2	30.6	71.5
			1.0	15.6	55.4	35.2	30.1	72.4
			10.0	16.5	56.2	34.2	29.4	77.2
1000	3500	1000	3 x 10 ⁻⁵	12.4	55.3	37.5	31.5	59.3
			3.5 x 10 ⁻³	13.0	51.1	34.9	29.6	70.7
			0.1	14.4	52.6	29.9	24.5	69.6
			1.0	17.9	53.8	34.3	30.0	69.6
			10.0	18.5	52.0	30.7	25.6	69.6
1000	10,000	72	3 x 10 ⁻⁵	26.2	83.4	69.8	64.2	76.2
		1000	3 x 10 ⁻⁵	14.9	53.7	32.7	28.7	55.3
		1000	3.5 x 10 ⁻³	15.2	55.3	33.5	29.1	69.1
			1.0	16.0	53.8	33.5	28.3	69.6
1200	1000	1200	3 x 10 ⁻⁵	13.3	34.6	47.1	20.8	50.4
			3.5 x 10 ⁻³	11.9	45.2	31.8	26.1	65.9
			0.1	13.0	48.9	31.9	26.8	61.8
			1.0	14.9	47.4	32.4	26.4	68.3
			10.0	16.8	48.8	33.4	26.4	67.5
1200	3500	1200	3 x 10 ⁻⁵	11.1	34.0	46.4	21.0	54.9
			3.5 x 10 ⁻³	10.6	42.7	31.9	26.3	61.8
			0.1	12.3	44.7	30.8	25.8	61.5
			1.0	14.0	46.3	30.7	25.6	64.2
			10.0	16.7	47.1	31.4	25.9	63.9
1200	10,000	72	3 x 10 ⁻⁵	26.2	86.3	63.6	59.4	64.2
		1200	3 x 10 ⁻⁵	12.8	34.3	46.8	22.3	65.0
		1200	3.5 x 10 ⁻³	12.2	44.0	28.4	23.6	55.3
			1.0	13.8	44.4	26.5	21.8	68.8

FIGURE HEDL-21. Effect of Strain Rate on Strength Properties of Thermally Aged 304 SS at 800°F.

FIGURE HEDL-22. Effect of Strain Rate on the Strength Properties of Thermally Aged 304 SS at 1000°F.

FIGURE HEDL-23. Effect of Strain Rate on Strength Properties of Thermal Aged 304 SS at 1200°F.

FIGURE HEDL-24. Effect of Strain Rate on the Ductility of Thermally Aged 304 SS at 800°F.

FIGURE HEDL-25. Effect of Strain Rate on the Ductility of Thermally Aged 304 SS at 1000°F.

FIGURE HEDL-26. Effect of Strain Rate on the Ductility of Thermally Aged 304 SS at 1200°F.

B. SUBCRITICAL CRACK GROWTH--INCOLOY 800

(Last Reported October-November-December 1973)

L. A. James/R. L. Knecht, J. J. Holmes

1. Objective

The objectives of this study are to determine the subcritical crack growth properties of FFTF/LMFBR structural materials for various operating environments, and to perform fatigue failure analyses for the vessel, primary coolant boundary components and other FFTF structures.

2. Accomplishments and Status

Linear-elastic fracture mechanics techniques were used to characterize the fatigue-crack propagation behavior of Incoloy 800. Two material heats were utilized representing material Grades 1 and 2 (mill-annealed and solution-annealed, respectively). The mechanical properties and chemical analyses are given in Tables HEDL-11 and 12, respectively. Since the objective of this study was to characterize the effect of temperature upon crack extension behavior, all elevated temperature tests were conducted at a constant cyclic frequency and stress ratio.

The results for the tests conducted over the range 75-1200°F are shown in Figures HEDL-27 through 31. Also shown are the results of Carden⁽⁵⁾ who tested Grade 1 material at 75°F and 1200°F. It will be noted that good agreement is obtained between the results of the present study and Carden's results. Comparison of Figures HEDL-27-31 reveals that the fatigue-crack growth rate increases with increasing test temperature. The increase, however, is considerably less than that observed for austenitic stainless steels tested under identical conditions.⁽⁶⁾ For example, at $\Delta K = 20,000 \text{ lb}/(\text{in.})^{3/2}$, the crack growth rate of Incoloy 800 increased by a factor of 4.1 between 75°F and 1200°F, while the crack growth of annealed Type 316 tested under identical conditions increased by a factor of 11.8 over the same temperature range.⁽⁵⁾ The reason for this difference in behavior is not known, but is thought to be related to the superior oxidation resistance of Incoloy 800.⁽⁶⁾

Tests conducted on the Heat A (Grade 2) material at 1200°F suggested the possible existence of a crack growth threshold at $\Delta K \approx 16,000 \text{ lb}/(\text{in.})^{3/2}$. At this temperature, cracks loaded below this level of ΔK did not propagate, even after the application of a great many cycles and crack extension would not proceed until the loads were increased such that ΔK exceeded 16,000 $\text{lb}/(\text{in.})^{3/2}$. Sufficient testing was not done to determine if this behavior was unique to Grade 2 material, or due to some other factor, but the phenomenon deserves further study.

Two specimens were aged in an air environment at 1000°F to determine if thermal aging (i.e., the precipitation of carbides and intermetallics) had any effect on the crack growth behavior. The results of these tests, shown in Figure HEDL-32, show that there is a very slight tendency for crack growth rates to decrease in the aged material. This is consistent with the results for several austenitic stainless steels.⁽⁷⁾ The decrease in crack growth rates of the aged stainless steels was attributed to a partial crack blunting and subsequent reinitiation as the crack encountered second-phase particles. Such a mechanism could also apply to the observed behavior in Incoloy 800.

TABLE HEDL-11
CHEMICAL COMPOSITION (PERCENT BY WEIGHT)

	C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	B
Heat A*	.07	.86	48.01	.007	.35	.18	30.29	19.26	.28	.31	-----
Heat B*	.03	1.05	46.48	.007	.26	.16	32.44	19.25	.10	.20	.0002
Carden***	.05	.95	45.28	.007	.36	.32	31.39	20.78	.38	.46	-----

*Huntington Heat No. HH2531A; ASME SB-409, Grade 2

**Huntington Heat No. HH7531A; ASME SB-409, Grade 1

***Huntington Heat No. HH2521A; ASME SB-409, Grade 1

TABLE HEDL-12
ROOM TEMPERATURE MECHANICAL PROPERTIES

	0.2% YS	Ult Strength	% Elong.	% Red. Area	Hardness (R _B)	ARTM Grain Size	Condition
Heat A*	27,000 psi 18.98 kg/mm ²	76,500 psi 53.76 kg/mm ²	53.0	----	69	1.5	Hot rolled, descaled, SA
Heat B*	35,5000 psi 24.96 kg/mm ²	75,500 psi 53.08 kg/mm ²	48.0	72.0	71	4.5	Hot rolled, descaled, Mill A
Carden***	42,500 psi 29.88 kg/mm ²	85,500 psi 60.12 kg/mm ²	39.0	----	76	8.0	Cold rolled, pickled, Mill A

*Huntington Heat No. HH2531A; ASME SB-409, Grade 2; 0.5 in. plate.

**Huntington Heat No. HH7531A; ASME SB-409, Grade 1; 1.0 in. plate.

***Huntington Heat No. HH2521A; ASME SB-409, Grade 1; 0.062 in. sheet.

3. References

6. H. L. Eiselstein and E. N. Skinner, "The Effect of Composition on the Scaling of Iron-Chromium-Nickel Alloys Subjected to Cyclic Temperature Conditions," ASTM STP-165, pp. 162-172, 1954.
7. L. A. James, "Effect of Thermal Aging Upon the Fatigue-Crack Propagation Behavior of Austenitic Stainless Steels," Metallurgical Transactions, Vol. 5, No. 4, 1974.

FIGURE HEDL-27. Fatigue-Crack Propagation Behavior of Incoloy 800 at 75°F (24°C).

FIGURE HEDL-28. Fatigue-Crack Propagation Behavior of Incoloy 800 at 600°F (316°C).

FIGURE HEDL-29. Fatigue-Crack Propagation Behavior of Incoloy 800 at 800°F (427°C).

FIGURE HEDL-30. Fatigue-Crack Propagation Behavior of Incoloy 800 at 1000°F (538°C).

FIGURE HEDL-31. Fatigue-Crack Propagation Behavior of Incoloy 800 at 1200°F (649°C).

FIGURE HEDL-32. Comparison of the Fatigue-Crack Propagation Behavior of Aged and Unaged Incoloy 800 at 1000°F (538°C).

C. SUBCRITICAL CRACK GROWTH--SA-351

(Last Reported January-February-March 1974)

L. A. James/R. L. Knecht, J. J. Holmes

1. Objective

The objectives of this study are to determine the subcritical crack growth properties of FFTF/LMFBR structural materials for various operating environments, and to perform fatigue failure analyses for the vessel, primary coolant boundary components and other FFTF structures.

2. Expected Achievements

This body of data represents completion of the initial experimental check on core basket materials performance. Further tests, on specimens included in the FTR surveillance subassemblies, will be conducted in order to evaluate the materials performance under conditions which are more closely representative of actual operating conditions. In addition, similar characterizations, as well as creep-rupture property evaluations, will be carried out on materials representing other FTR components as they are discharged from EBR-II subassemblies X-195A and X-198.

3. Accomplishments and Status

Cast austenitic stainless steels are employed in a number of structural applications (e.g., core support structures, valve bodies, pump housings and impellers, etc.) in FFTF/CRBP designs. Some of these components will be subjected to loadings in service that are cyclic in nature, and knowledge of the fatigue-crack growth behavior of cast stainless steels is necessary for design and safety analyses. Therefore, the present study characterized the effect of temperature upon the fatigue-crack growth behavior of one cast austenitic stainless steel: ASTM SA-351, Grade CF8 (procured to RDT M4-2T).

The specimens employed were of the ASTM "Compact Specimen" design, and had the planar dimensions corresponding to a one-inch thick specimen. The thickness was, however, approximately 0.51 inch. Two orientations of the crack, relative to the casting, were tested, and these were designated "A" and "B". The crack propagation direction in orientation "A" was perpendicular to that of orientation "B". Tests were conducted on an MTS testing machine using load as the control parameter. A sinusoidal waveform was employed, and the stress ratio R was 0.05 for all tests. A cyclic frequency of 40 cpm was used for all tests except at room temperature where frequency would not be expected to be an important parameter. Tests were conducted in an air-circulating furnace at 75, 600, 800, 1000 and 1200°F.

The experimental results are shown in Figures HEDL-33 through 37. Also given, for comparison, are scatter bands for annealed Types 304 and/or 316 SS tested under similar conditions. Several observations may be made concerning these results: 1) The fatigue-crack growth rate at a given value of ΔK increases with increasing test temperature. This is consistent with the behavior of other austenitic steels tested in an air environment. 2) In general, the crack growth rates in the cast stainless steel were slightly lower than those in annealed plate material tested under similar conditions. This is consistent with observations on Types 304/308⁽¹⁾ and 316/316⁽²⁾ weldments which also represented crack growth in a cast structure. 3) There does not appear to be any observable difference between the crack growth behavior for orientations "A" and "B". Another observation, not apparent in Figures HEDL-33 through 37, is that there was more crack branching observed (microscopically and macroscopically) in the

FIGURE HEDL-33. Fatigue Crack Propagation Behavior of ASME SA-351 Grade CF8 Cast Stainless Steel at 75°F (24°C).

FIGURE HEDL-34. Fatigue Crack Propagation Behavior of ASME SA-351 Grade CF8 Cast Stainless Steel at 600°F (316°C).

FIGURE HEDL-35. Fatigue Crack Propagation Behavior of ASME SA-351 Grade CF8 Cast Stainless Steel at 800°F (427°C).

FIGURE HEDL-36. Fatigue Crack Propagation Behavior of ASME SA-351 Grade CF8 Cast Stainless Steel at 1000°F (538°C).

cast material than normally observed in plate material. This is consistent with observations on crack growth in weldments,^(1,2) and may account for the slightly lower crack growth rates in the castings, relative to plate material.

FIGURE HEDL-37. Fatigue Crack Propagation Behavior of ASME SA-351 Grade CF8 Cast Stainless Steel at 1200°F (649°C).

4. References

1. L. A. James, "Crack Propagation Behavior in Type 304 Stainless Steel Weldments at Elevated Temperature," Welding Journal Research Supplement, Vol. 52, No. 4, April 1973, pp. 173s-179s.
2. P. Shahinian, H. H. Smith and J. R. Hawthorne, "Fatigue Crack Propagation in Stainless Steel Weldments at High Temperature," Welding Journal Research Supplement, Vol. 51, No. 11, November 1972, pp. 527s-532s.

D. FTR COMPONENT MATERIALS EVALUATION

J. M. Steichen/R. L. Knecht, J. J. Holmes

1. Objective

The objective of this program is to determine the mechanical properties of FTR component materials for anticipated service conditions. Included as an integral part of the program is the influence of both thermal history and fast neutron irradiation on observed behavior. Results of this work are used for design confirmation, operations support of FFTF and for optimizing materials selection and application for future LMFBRs.

2. Accomplishments and Status

The creep rupture properties of actual FTR primary piping (316H stainless steel, ~ 28-inch diameter) have been determined. Tests were performed on three heats of material at temperatures of 1050, 1200, and 1400°F and at stress levels to produce rupture in times to 2500 hours. Preliminary results from these tests are presented herein.

The Type 316H stainless steel FTR 28-inch diameter primary pipe was formed by the Rollmet process followed by a final anneal at 1850°F for 45 minutes and then force air cooled. Three heats of piping material were tested. The chemical composition and tensile properties for these heats have been reported previously.

All testing was performed in argon within recommended ASTM practice.

3. Results

Preliminary results of creep rupture tests at the three test temperatures for three heats of piping material are listed in Table HEDL-13 and illustrated in Figures HEDL-38 through 41. In Figure HEDL-38, stress versus rupture life for specimens fabricated from the longitudinal forming direction of the pipe for the three heats and the transverse direction for one heat (Heat 55320) are presented. A linear relationship between log stress and log rupture time is observed.

E. FFTF/LMFBR WELDMENT MATERIALS EVALUATION

A. L. Ward/R. L. Knecht, J. J. Holmes

1. Objective

The objective of this program is to describe the effects of elevated temperatures and fast reactor environments on the mechanical properties and microstructures of FFTF/LMFBR weldments and weld-deposited materials. Property changes due to neutron irradiation and high temperature microstructural instability in these materials are largely undefined. The purpose of this report is to present: 1) pre- and post-irradiation creep-rupture test results for weld-deposited CRE Type 308 which is representative of that used in the FTR pressure vessel fabrication; and 2) tensile test results for the same material following aging at 960°F for durations to 10,000 hours.

FIGURE HEDL-38. Stress-Rupture Behavior of 316H FTR Primary Piping.

FIGURE HEDL-39. Stress vs Minimum Creep Rate for 316H FTR Primary Piping.

FIGURE HEDL-40. Rupture Ductility of 316H FTR Primary Piping.

FIGURE HEDL-41. Comparison of Stress-Rupture Behavior of FTR 316H Primary Piping to ASME Code Case 1592.

TABLE HEDL-13
CREEP RUPTURE PROPERTIES OF FTR 316H PRIMARY PIPING

Heat No.	Temp., °F	Stress, ksi	Min. Creep Rate in/in/hr	Rupture Life, hr.	Elongation at Fracture, %
55320	1050	65	3.2×10^{-3}	23.4	42.9
		60	9.5×10^{-4}	91.5	34.9
		50	2.7×10^{-4}	360.5	27.3
		45	4.3×10^{-5}	2,483.2	28.3
55320-Trans.	1050	62	1.2×10^{-3}	33.8	41.3
		48	1.7×10^{-4}	502.6	22.0
55318	1050	60	1.1×10^{-3}	67.3	32.5
55319	1050	60	1.9×10^{-3}	60.5	38.9
		48	1.0×10^{-4}	> 512	> 24.0
55320	1200	40	7.7×10^{-3}	30.3	47.2
		34	1.8×10^{-3}	120.6	51.2
		30	3.9×10^{-4}	448.8	55.8
		26	2.1×10^{-4}	970.0	61.5
		24	9.4×10^{-5}	2,132.6	59.5
55320-Trans.	1200	35	2.9×10^{-3}	73.2	48.9
		29	3.9×10^{-4}	529.5	46.6
55318	1200	35	3.1×10^{-3}	76.9	47.7
		30	5.7×10^{-4}	290.5	51.1
55319	1200	35	2.6×10^{-3}	88.8	40.0
		30	4.5×10^{-4}	367.6	51.8
55320	1400	20	8.5×10^{-3}	19.5	68.2
		15	2.0×10^{-3}	121.5	67.7
		12	4.8×10^{-4}	548.1	> 69.5
55320-Trans.	1400	14	1.0×10^{-3}	222.7	74.0
55318	1400	14	1.6×10^{-3}	196.2	74.7
55319	1400	14	1.4×10^{-3}	222.3	80.0

2. Accomplishments and Status

Weld-deposited CRE 308 SS has been the subject of extensive study because of its use in the fabrication of the FTR pressure vessel and its potential use in the Demonstration reactor system. The unofficial CRE designation denoted "controlled residual elements", specifically boron, phosphorous and titanium, which were intentionally introduced into the weld metal through the flux coatings of electrodes using standard 308 core wires. Pre- and Post-irradiation (EBR-II: $0.18-0.55 \times 10^{22}$ n/cm², total at 900°F) creep-rupture tests at 1000°F and tensile tests (at 960°F) of specimens aged in static sodium at 960°F, the maximum vessel operating temperature for periods of 0, 100, 300, 1000, 3000 and 10,000 hours have been completed and the results are reported here. All of the specimens included in this study represent the chemical composition (Table HEDL-14) and weld process (SMA) used in the vessel and some (V2-specimens) were taken from strictly prototypic vessel weldments. Two types of weld deposits are represented in this work: 1) 1-1/4 inch thick weld-pad buildup from which all-weld transverse (to weld axis) specimens were taken (SET-); and 2) butt welds in 2-3/8 inch

Type 304 plate (SEL-, SEC, and V2-). Except for the SEC- specimens which are transverse composite (weld-base-HAZ) all specimens taken from the butt welds are longitudinal all-weld.

TABLE HEDL-14
CHEMICAL COMPOSITION OF FTR VESSEL WELD MATERIALS

Element	CRE 308 Lot CAEA (a)	CRE 308 Lot JAFA (b)	Type 304 Ht. 300188-1A (c)
C	.066	.060	.070
Mn	1.95	1.90	1.39
Si	.52	.37	.56
S	.016	.004	.016
P	.043	.044	.016
Cr	19.73	19.81	18.48
Ni	9.88	9.77	8.69
Mo	.25	.12	----
N	.039	.046	----
Cu	.16	.15	----
Co	.07	.17	.06
B	.007	.007	----
Ti	.06	.06	.015

(a) Lot CAEA represented by specimens with prefixes SET-, SEL-, and SEC-.

(b) Lot JAFA represented by specimens with prefix V2-.

(c) Type 304 Ht. 300188-1A included in composite specimens, SEC-.

3. Results

Pre- and post-irradiation creep-rupture results are listed in Table HEDL-15 and are presented graphically in Figures HEDL-42 through 45. Rupture life, minimum creep rate and total elongation were determined for all materials at 1000°F.

Rupture life results are presented on a conventional log stress-log rupture time plot in Figure HEDL-42. The dashed-line band represents the rupture life behavior of wrought 304 SS (minimum and average) as determined by Blackburn.⁽¹⁾ Rupture life values for the weld materials in general, unirradiated and irradiated, tend to be consistent with the average values for the base metal (304). The rupture lives of the weld-deposited materials seem to exhibit a slightly weaker stress-dependence than does the base metal, although the degree of data scatter precludes a definite conclusion. No consistent irradiation effect is detectable and it is clear that the degree of data scatter is greater than the irradiation effect on rupture life. The significance of this conclusion lies in the fact that the fluences represented here are greater, by factors ranging from 60 to 180, than the maximum expected in the FTR vessel wall. These data thus indicate that the effects of neutron irradiation on the vessel during its lifetime are negligible with regard to rupture life considerations.

FIGURE HEDL-42. Pre- and Post-Irradiation Stress vs Rupture Life Relationship for CRE 308 Weld Metal.

FIGURE HEDL-43. Larson-Miller Parameter as a Function of Initial Stress for CRE 308 Weld Metal at 1000°F.

FIGURE HEDL-44. Pre- and Post-Irradiation Stress vs Minimum Creep Rate Relationship for CRE 308 Weld Metal.

FIGURE HEDL-45. Pre- and Post-Irradiation Rupture Ductility of CRE 308 Weld Metal.

TABLE HEDL-15

PRE- AND POST-IRRADIATION CREEP-RUPTURE PROPERTIES OF CONTROLLED
RESIDUAL ELEMENTS (CRE) 308 WELD-DEPOSITED MATERIALS AT 1000°F

Specimen Identification (a)	Fluence, 10^{22} n/cm ²		Irrad. Temp.	Initial Stress, ksi	Rupture Time, hr.	Rupture Elong., %	Min. Cr. Rate %·hr ⁻¹	Larson-Miller Parameter (d)
	Total	E>0.1 MeV						
SET-3-17 (B06110)	---	---	---	50	55.6	21.6	.084	31747
SEL-3-37 (B06128)	---	---	---	45	217.9	22.8	.036	32613
SEL-3-21 (B06129)	---	---	---	43	350.9	18.4	.023	32915
SET-3-3 (B06130)	---	---	---	40	9761.9	13.3	.00024	35025
S-3-3 (B06134)	---	---	---	35	>3460	(b)	.00011	---
V2-3-5 (B06159)	---	---	---	45	152.1	17.9	.047	32385
V2-3-7 (B06163)	---	---	---	45	127.3	25.4	.078	32272
V2-3-8 (B06132)	---	---	---	40	3061.9	10.2	.00088	34289
V2-3-3 (B06165) (c)	---	---	---	45	77.4	20.3	.088	31957
V2-3-11 (B06166)	---	---	---	45	1232.0	10.0	.0016	33711
SEC-2-24 (B06142)	---	---	---	55	18.0	14.0	.15	31032
SET-2-3 (B06143)	---	---	---	45	649.9	8.4	.0039	33206
SEC-2-25 (B06144)	---	---	---	40	2010.9	6.7	.0011	34022
SEC-4-28 (B06146)	---	---	---	38	>2018	(b)	.00016	---
SET-01 (B06117)	.55	.48	900	55	6.9	10.6	.387	30424
SET-02 (B06121)	.55	.48	900	50	66.9	>5.2	.021	31865
SET-04 (B06155)	.55	.48	900	45	903.5	7.2	.0014	33515
SET-05 (B06188)	.55	.48	900	40	>12625.4	(b)	.000063	35187
SET-10 (B06119)	.18	.16	900	55	11.2	13.1	.396	30732
SET-13 (B06156)	.18	.16	900	50	30.5	>16.5	.24	31115
SET-11 (B06131)	.18	.16	900	45	412.8	13.2	.011	33018
SET-12 (B06150)	.18	.16	900	40	2457.8	7.1	.00094	34149
SEC-01 (B06123)	.55	.48	900	55	33.2	2.8	.023	31420
SEC-04 (B06160)	.55	.48	900	50	117.3	4.1	.0097	32221
SEC-02 (B06132)	.55	.48	900	45	~503	2.3	.0015	33144
SEC-03 (B06151)	.55	.48	900	40	887.8	2.4	.00038	33504
SEC-10 (B06120)	.18	.16	900	55	24.2	11.6	.080	31220
SEC-13 (B06161)	.18	.16	900	50	52.4	9.8	.049	31710
SEC-11 (B06133)	.18	.16	900	45	213.3	3.6	.0050	32600
SEC-12 (B06153)	.18	.16	900	40	576.0	5.8	.0010	33229

(a) SET: Transverse-weld pad; SEL: Longitudinal-butt weld; SEC: Transverse-butt weld;

V2: Longitudinal-butt weld.

(b) Test interrupted prior to rupture.

(c) Specimen taken from highly strained center of weldment.

(d) L-M Parameter = $(T+460)(20+\log t_r)$ where $T=1000^\circ\text{F}$ and t_r =rupture time (hours).

In order to compare the rupture life results with data generated by ORNL⁽²⁾ on representative CRE 308 weld metal, the data are presented in Figure HEDL-43 by the Larson-Miller time-temperature parameter technique. The Larson-Miller parameter, $(T + 460)(20 + \log t_r)$, compensates for test temperature differences which, in the case of the ORNL data, range from 900 to 1200°F. The ORNL trend curve (Figure HEDL-43) is clearly supportive to the rupture time data in the present work and thus to the conclusions given above. It should be noted that the ORNL work included specimens taken from various locations through the thickness of the weldment. The two trend curves given in Figure HEDL-43, labeled L1 and L2, L3 represent rupture life behavior of specimens taken from near the surface (L1) and from locations approximately 0.6 to 0.1 inch below the surface (L2, L3), respectively. Specimens utilized in the present work correspond approximately to the ORNL L2 location index.

Minimum creep rate results are presented in conventional log stress-log minimum creep rate plots in Figure HEDL-44. Again, the 304 SS base metal behavior is represented by a curve from the Blackburn analysis.⁽¹⁾ In general, the weld metal minimum creep rate values compare favorably with the 304 curve at high stresses and less closely at low stresses. Although the data scatter is great, there does appear to be a trend toward lower creep rates with increasing

fluence in all weld materials represented. The tendency toward lower creep rates at lower stresses lends additional confidence in the satisfactory performance of these materials in service.

Total rupture elongation values are given in Figure HEDL-45. The most distinctive feature of these data is the irradiation-induced loss in creep ductility. Possible reasons for the losses include: 1) helium embrittlement, which in most austenitic stainless steels, would be expected to have similar effects at somewhat higher test temperatures; 2) embrittlement by transformation of second phase delta-ferrite to sigma phase; and 3) a boundary sliding deformation mode which results in the concentration of strains at grain and interphase boundaries causing premature failure. The fact that these ductility losses had no apparent effect on the rupture life suggests that the losses are compensated for, to some extent, by the lower minimum creep rates and, in any case, would not be expected to impose a significant service performance penalty.

TABLE HEDL-16
TENSILE PROPERTIES OF AGED (960°F) CRE 308 WELD DEPOSITS PROTOTYPIC OF
FTR PRESSURE VESSEL FABRICATION WELDMENTS AT 960°F

Specimen Identification	Aging Time (hr)	Strength (ksi)			Elongation (%)		
		PEL	Yield	UTS	Uniform	Total	RA (%)
V2-X2-15 (H08265)	0	29.5	36.2	60.3	24.2	28.1	59
V2-H3-15 (H08266)	0	33.3	40.0	58.9	18.0	23.0	62
V2-X2-5 (H08261)	100	34.3	40.8	59.4	16.4	18.6	49
V2-H3-5 (H08262)	100	37.5	45.6	59.4	16.1	19.7	56
V2-X2-6 (H08263)	300	36.1	43.5	57.3	13.7	16.5	52
V2-H3-6 (H08264)	300	35.4	41.5	56.7	15.6	18.9	46
V2-W2-6 (H08296)	1000	33.9	41.6	55.3	12.5	15.1	52
V2-H3-7 (H08293)	1000	36.7	39.7	56.8	15.0	18.5	49
V2-X2-7 (H08294)	1000	35.7	43.0	57.2	10.6	14.0	44
V2-X2-8 (H08297)	3000	42.6	47.8	57.2	7.1	9.9	40
V2-H3-8 (H08298)	3000	41.8	48.4	58.0	6.9	10.5	43
V2-H3-9 (H08312)	10,000	39.0	50.2	59.7	7.7	10.5	50
V2-X2-9 (H08311)	10,000	32.5	46.1	56.4	9.2	11.8	52

The tensile properties of CRE Type 308 weld metal (V2-only) following aging at 960°F are listed in Table HEDL-16 and plotted as functions of aging time in Figures HEDL-46 (strength) and 47 (ductility). These data represent a test temperature of 960°F and a conventional strain rate, 0.018 min^{-1} . Ultimate tensile strength values remain nearly unchanged over the entire range of aging times, 0 to 10,000 hours. A very slight decrease to a minimum at 1000 to 3000 hours may or may not represent a real aging effect. Larger changes are evident in the PEL and yield strength data. A slight increase from 0 to 100 hours is maintained to about 1000 hours, where an additional increase between 1000 and 3000 hours occurs. Between aging times of 0 and 3000 hours, all of the ductility parameters decrease continuously (Figure HEDL-47). The minimum at ~ 3000 hours suggests that the properties are responding to

HEDL-57

FIGURE HEDL-46. Strength of Aged CRE 308 Weld Metal at 960°F.

FIGURE HEDL-47. Tensile Ductility of Aged CRE 308 Weld Metal at 960°F.

some type of time-temperature dependent overaging phenomenon, although there is no other evidence available to support this view. In an aging study at ORNL ⁽³⁾ it was found that 4000 hours at 900°F produced only minor decreases in the uniform and total elongation values and 4000 hours at 1100°F resulted in essentially no change in ductility. Additional work on similar materials has shown that the tensile properties are essentially unchanged over the time range from 1000 to 10,000 hours at 1000°F. Studies of the microstructures associated with the subject aging times are in progress at HEDL and at ORNL.

4. Expected Achievements

During the next reporting period tensile and creep-rupture characterizations of representative and prototypic FTR piping weld materials (16-8-2/316H and 308/304H) will continue. In addition tensile tests will be conducted on large vessel weld sections for the purpose of defining the strain distributions in the entire weldments as opposed to weld, HAZ, or base metals. Post-irradiation tensile results are also expected to be available for weld-deposited Type 308 which is prototypic of the FTR core basket and core support structure weldments.

5. References

1. L. D. Blackburn, "Isochronous Stress-Strain Curves for Austenitic Stainless Steels," The Generative of Isochronous Stress-Strain Curves, Ed. A. O. Shaefer, American Society of Mechanical Engineers, New York, Nov 1972, pp. 15-48.
2. R. T. King, J. O. Stiegler and G. M. Goodwin, "Creep Properties of a Type 308 Stainless Steel Pressure Vessel Weld With Controlled Residual Elements," ORNL-TM-4131, May 1973.
3. R. T. King, E. Bolling and B. McNabb, Jr., "Tensile Properties of Aged SMA Type 308 CRE Stainless Steel FFTF Vessel Test Welds," ORNL-4936, October 1973, p. 133.

III. CLADDING DEVELOPMENT AND PROCESS

L. H. Rice

A. FUEL CLADDING CHARACTERIZATION

(Last Reported December 1973)

M. M. Paxton/R. L. Knecht, J. J. Holmes

1. Objective

The program objective is to define the mechanical properties, metallurgical structure, chemistry and metallurgical stability of prototypic and production FTR fuel cladding purchased under the cladding development program. Cladding from this program is utilized for vendor qualification, FFTF development programs and FTR core loadings.

2. Accomplishments and Status

Scope of Work - The recovery and recrystallization characteristics of one 20% CW 316 SS tubing series (N-Lot) have been established and reported.⁽¹⁾ The recovery and recrystallization characteristics of eleven prototypic and production fuel cladding tubing lots at 1400, 1500 and 1600°F have been determined to establish lot-to-lot variations and to provide a basis for possible improvements in cladding recrystallization characteristics.

3. Results

Material and Procedure - Eleven lots of 20% CW 316 SS tubing (N, O, R, T, U, X, BB, CN-13, CN-17, P and S) were utilized in this study. Isothermal annealing curves were

for each tube by aging four-inch tubular specimens in helium at 1400 to 1600°F for times from 0.1 to 2000 hours. The aged specimens were subsequently tensile tested in vacuum (5 to 10 microns) at 1000°F at a strain rate of 0.01 min^{-1} utilizing an Instron Tensile Machine. Specimens were gripped by using a compression fitting technique.⁽²⁾

Results - The 0.2% yield stress at 1000°F was used as the measure of the annealing effects of time at temperature. The resulting isothermal annealing curves at 1500 and 1600°F (Figures HEDL-48 and 49) clearly show that the recrystallization time at these two temperatures can vary significantly from lot to lot. At 1600°F, the recrystallization time ranges from a low of ≤ 0.2 hours to a high of 7 hours. At 1500°F, the range is 10 hours to 200 hours.

Because the variations in recrystallization times were greater than would be expected from data scatter, an attempt was made to correlate the variations to some variable that exists between the tube lots. Correlations with respect to chemistry variations,⁽³⁾ drawing sequence, drawing technique, melting practice were all unsuccessful. However, the recrystallization time for a temperature was found to correlate quite well with the heat treatment temperature used prior to the final 20% reduction in area. This correlation is shown in Figure HEDL-50 for the seven tube lots for which there is sufficient processing data.

These data show that some thermally activated process which occurs during the heat treatment before the final draw has a pronounced effect on the subsequent recrystallization kinetics. No detailed in-depth study was attempted using these data due to lot-to-lot variations in chemistry, time at temperature during heat treatment, exact cold work level, etc. However, an experiment using a series of tubes processed in a commercial tube mill under well characterized conditions is now underway. Twenty-one tubes were selected from a single processing lot (consisting of ≈ 200 tubes) just prior to the final anneal and three tubes were given each of the following heat treatments:

Temperature (°F)	Furnace Feed Rate	Time at Temperature (Peak $\pm 5^\circ\text{F}$)
1850	36 in/min	7-10
1900	36 in/min	7-10
1950	36 in/min	7-10
2000	36 in/min	7-10
2050	36 in/min	7-10
1950	48 in/min	2-4
2050	48 in/min	2-4

These tubes were subsequently given a 20% reduction in area sequentially using the same dies to minimize or eliminate any change in cold work level due to die wear.

4. Expected Accomplishments

During the next reporting period, isochronal annealing curves will be completed at 1400, 1500 and 1600°F on these special tubes. In addition, an effort will be made to determine what effect the various heat treatments have had on the metallurgical condition of the annealed and cold-worked tubing.

FIGURE HEDL-48. Isothermal Annealing Curves at 1600°F for Eleven Prototypic FTR Fuel Cladding Tubing Lots.

FIGURE HEDL-49. Isothermal Annealing Curves at 1500°F for Eleven Prototypic FTR Fuel Cladding Tubing Lots.

FIGURE HEDL-50. A Correlation of Heat Treatment Temperature Prior to the Final 20% Reduction in Area and Recrystallization Time at 1500 and 1600°F.

B. REFERENCES

1. M. M. Paxton and J. J. Holmes, "Recovery and Recrystallization of Prototypic FTR Fuel Cladding--20% Cold Worked 316 Stainless Steel Tubing," Westinghouse Hanford Company, HEDL-TME 71-126, September 1971.
2. M. M. Paxton, "Elevated Temperature Tensile Grips for Tubing," ASTM Elevated Temperature Testing Problem Areas, STP 488.
3. M. M. Paxton and J. L. Straalsund, "The Effect of Chemistry Variations on the Recrystallization Temperature of 20% Cold Worked Type 316 Stainless Steel," Westinghouse Hanford Company, HEDL-TME 72-75, June 1972.

IV. IRRADIATION-INDUCED SWELLING OF 316 SS

A. SWELLING OF CW 316 SS IRRADIATED TO HIGH NEUTRON FLUENCES

H. R. Brager/J. J. Laidler

1. Objective

The objective of this work is to determine the effect of high neutron fluence and irradiation temperature on the swelling behavior and microstructural stability of CW 316 SS, providing experimental data for updating and refinement of swelling design equations.

2. Accomplishments and Status

Specimens of 20% CW 316 SS were extracted from unstressed drawn tubes which were all irradiated in EBR-II to neutron fluences of about 9×10^{22} n/cm² ($E > 0.1$ MeV) as part of G.E.'s subassembly X-098/X-098A. Immersion density measurements of the nominally 40 milligram specimens have been completed, and a preliminary investigation of the microstructure of the specimens was conducted using the 1 MeV electron microscope.

Figure HEDL-51 shows the swelling, based on immersion density data, of the X-098 specimens plotted as a function of irradiation temperatures. The 2σ variation was calculated for specimens for which there were four separate density measurements. All other swelling values are based on two or three density measurements. The peak swelling was found in the specimens irradiated at a calculated temperature of 610°C. (the irradiation temperature calculations made available by H. Busboom, GE-BRO). The bold solid line is a reasonably close fit to the raw data. The thin dot-dash line represents a possible interpretation of the same data. Both curves are compared to recent French swelling data for 25% CW 316 SS.* The X-098 data indicate swelling from about one-half to the same as the French data, but peaking at more than 50°C higher temperature. The profilometry data (made available by H. Busboom, GE-BRO) in Figure HEDL-52 show that the maximum diameter increase in the X-098 specimens occurred at about 610°C for both solution-treated and cold-worked steel. The French data* showed that the peak swelling of solution-treated and CW 316 SS also occurred at one temperature, in that case, about 525°C. Although the exact chemical composition of the steel used by the French is not known, the concentration ranges of the alloying elements in the French steel are essentially identical to the ranges in commercially obtainable AISI 316 and bracket the elemental chemical composition of the steel used in X-098. The carbon concentration of the 316 used was about 0.05%, which is the same as that of the FTR first-clad material. It is believed that the difference in peak swelling temperature in the 0% and 25% CW 316 SS used by the French and in X-098 is not due to the expected differences in chemistry or manufacturing variables. The most likely explanation for the difference in peak swelling temperature is the variation in gamma heating values and analytical models used in calculating the irradiation temperatures. There is no clear-cut method of determining whether the French or the X-098 peak swelling temperature are "correct". Except for low neutron fluence specimens, specifically XX03, irradiation temperatures of EBR-II specimens examined to date are based on calculated values.

Figure HEDL-53 shows the neutron fluence at which voids are first observed in cold worked 316 SS, as a function of irradiation temperature. The previous lower-fluence data, of less than 5.3×10^{22} n/cm², had shown that void formation was inhibited at irradiation temperatures near 500°C. The new data indicate that, at some neutron fluence of greater than about 5.5×10^{22}

*These data were provided by J.N. Dupouy of the French C.E.N. Saclay in October 1973.

and less than $8.5 \times 10^{22} \text{ n/cm}^2$, void formation occurs over the entire irradiation temperature range from 370 to nearly 700°C.

FIGURE HEDL-51. Immersion density data of metal swelling in 20% CW 316 SS (X-098) irradiated in the EBR-II to a $\sim 9 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) as a function of irradiation temperature. The maximum swelling of about 2% occurs at a calculated temperature of 610°C.

Swelling observed in X-098 specimens, based on immersion density data, is compared with the current Stress-Free Swelling Design Equation in Figure HEDL-54. The present data indicate that the maximum swelling is less than that predicted by the Design Equation. Considering the bilinear swelling behavior of irradiated steels, the difference between the immersion density data and the Design Equation could be due to differences in the estimated incubation fluence or in the swelling rate. Electron irradiation results reported in previous HEDL Technical Progress Reports indicate that compositional variations in 316 stainless steel have little effect on the steady-state swelling rate. The incubation fluence, on the other hand, appears to be very sensitive to composition or fabrication variables. It is therefore quite likely that the difference between the swelling in the X-098 steel and that predicted by the current design equation (which is based on N-102 material) is due to variations in the incubation fluence.

Figure HEDL-52. Metal swelling of solution treated and 20% CW 316 SS from x-098 tubing as a function of irradiation temperature. The swelling values are based on profilometry measurements with the maximum diameter increase of both solution treated and 20% CW specimens occurring at the same temperature.

Table HEDL-17 summarizes the immersion density measurements and the preliminary TEM data. The swelling data from specimens irradiated at the lowest (450°C) and highest (690°C) temperatures are consistent with previous results on specimens which received comparably low neutron fluences (about $5 \times 10^{22} \text{ cm}^{-2}$). The 450°C irradiated specimen contained a moderately high density of voids which were fairly uniformly distributed throughout the matrix. The small volume of the observable voids was similar to the change in immersion density.

The specimens irradiated at 535°C and 610°C each contained unusual microstructural features. The void formation in the 535°C irradiated steel was very heterogeneous. Most of the steel contained a low density of voids; however, there were a few regions that contained a high concentration of large voids. These regions having large amounts of swelling were typically in the form of bands similar to that of microtwins. Figure HEDL-55 is a micrograph which shows one of these unusual bands of voids. The void volume in the banded region of this micrograph was 2.0%, compared to the immersion density change of 0.42%. The voids in the banded region were over twice as large and nearly 10 times as numerous as those found in the more typical low swelling regions. A quantitative comparison of the preliminary TEM data is shown in Table HEDL-17. The high void volume regions also contain a very high concentration of small precipi-

tates and appear to be bounded by regions which have a very high dislocation density. The void formation might have been enhanced by localized compositional variations and/or stresses.

TABLE HEDL-17
SUMMARY OF X-098 SPECIMEN IRRADIATION CONDITIONS, SWELLING INFORMATION AND
PRELIMINARY TRANSMISSION ELECTRON MICROSCOPY DATA.

SPECIMEN #	VOID DIAMETER (Å)			VOID NUMBER DENSITY (VOIDS cm^{-3})	VOID VOLUME $\Delta V/V$ (%)	SWELLING $\Delta \rho/\rho$ (%)	Φt $10^{22} \text{ n cm}^{-2}$ ($E > 0.1 \text{ MeV}$)	T_{irrad} ($^{\circ}\text{C}$)
	MIN.	MEAN	MAX.					
LP2C2D4	117	240	400	2.2×10^{14}	0.18%	0.13%	8.6	454
LP2G1D2	117	347	834	8.6×10^{12}	0.03%	0.42%	8.9	535
	117	749	1450	6.8×10^{13}	2.0%			
LP2K1D2	133	315	700	4.6×10^{13}	0.08%	2.30%	8.8	609
	467	1506	2767	1.1×10^{13}	3.63%			
LP2R1D2	---	---	---	$< 10^{12}$	0.0%	-0.009	9.4	689

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The steel irradiated at 610°C , which swelled about 2.3%, exhibited a duplex void size distribution, as illustrated in Figure HEDL-56. The void volume of the smaller sized voids having a mean void diameter of $\sim 315 \text{ Å}$ amounted to less than 5% of the immersion density change. A low number density of extremely large voids, mean diameter $\sim 1500 \text{ Å}$, were also produced; this void volume accounted for nearly all of the swelling. All of the large voids were attached to polyhedral precipitates identified to be M_{23}C_6 . The formation of large voids on precipitates might be due to a stress field attraction of vacancies to the precipitates or to dragging of vacancies to the precipitates or to dragging of vacancies to the precipitates. The micro-structure of voids associated with large M_{23}C_6 precipitates in this unstressed stainless steel is not unlike results reported in 1969 by U.K. investigators for neutron irradiated CW M316 cladding, fuel pin No. A 1064.*

*K. Q. Bagley, Et. al. "Voids formed by irradiation of reactor materials", proceedings B.N.E.S. Conference, Reading, Eds. S. S. Pugh, W. H. Loretta, D. I. A. Norris, March 24, 25, 1971, Pg. 6.

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FIGURE HEDL-53. Irradiation temperature and "incubation" neutron fluence for void formation in CW 316 SS.

FIGURE HEDL-54. The immersion density data of 20% CW 315 SS from X-098 compared with the current "nominal and "minimum" Stress-Free Swelling Design Equation.

FIGURE HEDL-55. A HVEM Bright Field Micrograph of 20% CW 315 SS Irradiated at 535°C to 8.9×10^{22} n/cm² ($E > 0.1$ MeV). The micrograph illustrates a non-typical area containing a banded region where a high density of large voids were formed which had a void volume of ~2%.

FIGURE HEDL-56. The Same 20% CW Steel as Figure 55 except Irradiated at $\sim 600^{\circ}\text{C}$. The voids were fairly homogeneously distributed, with the small voids not associated with precipitates.

B. IRRADIATION-INDUCED SWELLING VARIATIONS
RESULTING FROM COMPOSITIONAL MODIFICATIONS OF

316 SS

J. F. Bates

1. Introduction

The phenomenon of void swelling in stainless steels has received considerable attention due to the technological importance of these materials. The efficiency of LMFBR reactors can be greatly enhanced by reductions in the extent of swelling.

The alloy content of austenitic stainless steel is known to exert a strong influence on the swelling characteristics of the alloy. Consider, for example, the reduction in swelling rate observed in 316 SS relative to that in 304 SS. While there are gross compositional differences between these two alloys [304: Fe-19Cr-10Ni; 316: Fe-17Cr-12Ni-2.5Mo], there are also more subtle compositional variations, and the origin of the improved swelling behavior of 316 has not yet been established.

The observations reported here represent initial results of a major experimental program which has as its primary goal the identification of the optimum (based on swelling and mechanical properties) alloy and residual element content, of Type 316 SS. The program involves neutron and electron irradiation experiments and subsequent swelling determinations on alloys representing systematic, independent variations from the base composition of 316 SS.

2. Experimental Details and Techniques

The different alloys outlined in Table HEDL-18 are from special single-vacuum melted heats. Detailed fabrication history is contained in Reference 1 and will not be repeated herein. Except where specifically noted, the results refer to material in the solution annealed (1925°F/WQ) condition.

Densities were measured by means of the Archimedeian technique, before and after irradiation. Accuracy with this technique ranges from $\pm 0.02\%$ for these densities determined before irradiation to $\pm 0.05\%$ for these densities determined remotely, after irradiation. Swelling values are thus reported as the percent change in density based on the initial unirradiated values shown in Table HEDL-18.

Each of the alloys was analyzed for ferrite content, which was determined by means of a Severn Ferrite Indicator. Uncertainty in these determinations may run as high as $\pm 1\%$ by volume. None of the alloys registered over 0.5% ferrite, the lower detectable limit of the Severn technique, with the exception of Alloy 40. Alloy 40, a low chromium, low nickel alloy, registered 0.5%-1% ferrite, which is still within the scatter of the Severn Indicator.

3. Results

Table HEDL-19 is a complete summary of the swelling data for the annealed alloys. There is one point which must be kept in mind during the discussion of these data: They all represent low fluence ($2-4 \times 10^{22}$ n/cm² E>0.1 MeV) swelling. It is very probable therefore that they are not indicative of swelling behavior at higher fluences ($>10^{23}$ n/cm²). This can best be understood by reference to Figure HEDL-57, which shows expected swelling behavior for 20%

CW 316 SS. The swelling is typically characterized by an incubation or nucleation period and a growth period modeled by a bi-linear type of equation. Both the incubation time and the rate of swelling during the growth period may differ for elemental alloy modifications in such a manner that the relative swelling obtained for two alloys irradiated to a low fluence would not be indicative of the relative swelling obtained for these same two alloys at a higher fluence.

TABLE HEDL-18
METALLURGICAL VARIABLES ALLOY COMPOSITIONS

ALLOY NO	DENSITY (g/cm ³)	ELEMENT (WEIGHT %)											
		C	Mn	Si	P	S	Cr	Ni	Mo	Cu	Co	B	N
CARBON-NITROGEN VARIATIONS													
1	7.98131	0.003	0.92	0.40	0.009	0.008	16.95	12.01	2.35	0.10	0.11	0.0011	0.007
3	7.96941	0.047	0.94	0.39	0.010	0.007	16.99	12.05	2.33	0.10	0.12	0.0008	0.050
4	7.963	0.127	0.96	0.33	0.012	0.008	17.06	12.08	2.32	0.10	0.11	0.0010	0.006
5	7.95223	0.128	0.97	0.43	0.011	0.008	17.00	12.10	2.34	0.10	0.11	0.0008	0.111
45	7.9684	0.012	1.12	0.41	0.010	0.007	16.98	12.12	2.31	0.10	0.11	0.0012	0.13
PHOSPHORUS-SULFUR-BORON VARIATIONS													
6	7.96666	0.048	0.92	0.35	0.001	0.004	16.89	11.99	2.36	0.12	0.12	0.0005	0.054
7	7.96796	0.046	0.96	0.37	0.003	0.007	17.06	12.13	2.35	0.10	0.11	0.0005	0.052
8	7.96576	0.046	0.97	0.38	0.003	0.018	17.14	12.14	2.32	0.11	0.11	0.0005	0.054
9	7.96163	0.045	0.96	0.37	0.004	0.036	17.09	12.17	2.32	0.11	0.11	0.0005	0.055
10	7.96973	0.047	0.93	0.37	0.011	0.004	17.09	12.04	2.35	0.11	0.11	0.0005	0.052
11	7.96779	0.045	0.95	0.39	0.021	0.005	16.95	12.03	2.31	0.11	0.12	0.0005	0.054
12	7.96583	0.046	0.93	0.36	0.039	0.004	17.01	12.12	2.33	0.11	0.11	0.0005	0.057
13	7.96495	0.051	0.93	0.40	0.003	0.004	16.95	12.09	2.34	0.11	0.11	0.0012	0.057
14	7.96735	0.046	0.98	0.39	0.003	0.004	17.09	12.10	2.31	0.11	0.11	0.0030	0.054
15	7.96661	0.047	0.96	0.39	0.004	0.004	17.05	12.14	2.32	0.10	0.11	0.0057	0.052
MANGANESE VARIATIONS													
16	7.97462	0.045	0.01	0.43	0.011	0.008	16.92	12.15	2.45	0.10	0.11	0.0011	0.047
17	7.96861	0.046	0.92	0.38	0.009	0.007	17.00	12.07	2.33	0.10	0.11	0.0012	0.052
18	7.9617	0.047	2.04	0.38	0.010	0.008	17.17	11.91	2.21	0.10	0.11	0.0016	0.054
19	7.95103	0.044	3.89	0.37	0.009	0.007	17.03	11.98	2.30	0.10	0.11	0.0012	0.056
COPPER VARIATIONS													
20	7.96913	0.048	0.96	0.40	0.010	0.008	16.92	12.07	2.32	0.01	0.11	0.0010	0.049
22	7.96936	0.044	0.94	0.38	0.009	0.007	16.96	12.04	2.35	0.99	0.11	0.0012	0.052
23	7.97318	0.044	0.95	0.35	0.010	0.008	16.96	12.27	2.35	2.94	0.11	0.0012	0.052
24	7.9758	0.044	0.95	0.39	0.011	0.008	17.00	11.91	2.35	4.47	0.11	0.0013	0.055
SILICON VARIATIONS													
25	7.99698	0.046	0.95	0.01	0.010	0.008	16.88	11.98	2.31	0.10	0.11	0.0011	0.052
26	7.96249	0.045	0.94	0.48	0.009	0.008	17.00	12.12	2.34	0.11	0.11	0.0010	0.054
27	7.92499	0.045	0.94	0.95	0.011	0.007	17.06	12.19	2.35	0.10	0.11	0.0007	0.049
28	7.88573	0.044	0.93	1.47	0.010	0.008	17.07	12.30	2.38	0.10	0.11	0.0007	0.054
29	7.85047	0.045	0.94	1.96	0.010	0.007	17.13	12.39	2.41	0.11	0.11	0.0010	0.050
MOLYBDENUM VARIATIONS													
30	7.9363	0.044	0.99	0.40	0.010	0.007	16.89	12.31	0.01	0.10	0.11	0.0011	0.049
31	7.93893	0.041	0.98	0.38	0.010	0.007	16.86	12.04	0.11	0.10	0.11	0.0011	0.048
32	7.94894	0.045	0.99	0.40	0.010	0.008	16.90	11.99	1.00	0.10	0.11	0.0012	0.049
33	7.97632	0.047	0.92	0.38	0.011	0.008	16.96	12.09	3.03	0.10	0.11	0.0012	0.049
34	7.9536	0.045	0.87	0.40	0.011	0.008	17.09	12.24	4.93	0.10	0.11	0.0010	0.051
COBALT VARIATIONS													
35	7.96879	0.044	0.96	0.41	0.010	0.007	16.89	12.03	2.31	0.10	0.02	0.0010	0.049
37	7.97283	0.046	0.91	0.41	0.009	0.009	16.97	11.89	2.29	0.10	0.50	0.0010	0.051
39	8.00137	0.042	0.90	0.38	0.011	0.008	17.00	12.04	2.30	0.10	4.45	0.0013	0.049
CHROMIUM-NICKEL VARIATIONS													
40	7.9833	0.045	0.95	0.40	0.010	0.007	14.86	9.96	2.29	0.10	0.12	0.0013	0.050
41	7.99177	0.046	0.96	0.38	0.009	0.007	14.89	12.01	2.35	0.10	0.11	0.0013	0.052
42	7.99984	0.046	1.00	0.39	0.009	0.008	15.13	14.04	2.32	0.10	0.11	0.0008	0.048
43	7.96931	0.047	0.93	0.40	0.009	0.007	16.85	12.12	2.35	0.10	0.11	0.0005	0.052
44	7.9415	0.041	0.90	0.41	0.011	0.007	18.97	12.11	2.35	0.10	0.11	0.0010	0.049

TABLE HEDL-19. SWELLING DATA FOR ANNEALED METALLURGICAL VARIABLE ALLOYS

Alloy No.	% $\frac{\Delta\rho}{\rho}$	Pin-Level	ϕt , 10^{22} n/cm ² (E>0.1 MeV)	Temp. (°F)
<u>Carbon-Nitrogen</u>				
1	-.58	44-16	2.59	744-803
3	-.32	46-16	2.63	719-777
45	-.52	45-16	2.67	730-789
5	-.23	48-16	2.67	755-813
4	-.24	47-16	2.63	722-782
4	+.089	47-8	3.77	1057-1276
4	+.064	47-14	3.48	1095-1206
5	+.037	48-8	3.84	1075-1288
5	+.020	48-14	3.54	1086-1176
4	+.11	47-9	4.00	1315-1484
5	+.08	48-9	4.07	1326-1494
4	-.015	47-5	2.63	913-1001
5	-.044	48-5	2.67	930-1015
1	-.49	44-5	2.59	911-1001
45	-.11	45-5	2.67	891-978
3	-.044	46-5	2.63	909-995
3	+.050	46-14	3.47	1058-1169
1	-.097	44-14	3.53	1033-1146
3	+.081	46-9	4.00	1343-1506
3	+.066	46-8	3.77	1289-1466
1	+.020	44-8	3.86	1062-1272
45	+.021	45-8	3.84	1054-1272
45	+.058	45-9	4.07	1314-1484
45	-.044	45-14	3.54	1055-1169
1	+.053	44-9	4.11	1325-1493
<u>Sulphur</u>				
9	-.31	45-17	2.31	722-728
9	-.22	45-4	2.31	883-930
<u>Phosphorus</u>				
6	-.31	44-17	2.20	714-747
10	-.31	44-17	2.20	714-747
11	-.31	44-17	2.20	714-747
12	-.31	44-17	2.20	714-747
6	-.31	44-17	2.20	714-747
12	-.037	47-17	2.27	742-775
12	-.037	47-17	2.27	742-775
12	-.046	47-4	2.27	866-945
6	-.25	44-4	2.20	870-918
6	+.029	44-10	4.24	1318-1478
6	+.029	44-10	4.24	1318-1478
<u>Silicon</u>				
25	-.19	48-18	1.97	744-761
26	-.054	48-18	1.97	744-761
27	+.094	↓	↓	↓
28	+.094			
28	+.15			
29	+.15			
25	-1.19	49-14	3.53	1008-1128
26	-.061	↓	↓	↓
27	-.090			
28	-.19			
29	-.18			
25	-.48	48-3	1.97	863-889
26	-.077	↓	↓	↓
27	+.0001			
28	+.035			

TABLE HEDL-19 (Cont'd)

Alloy No.	% $\frac{\Delta\rho}{\rho}$	Pin-Level	ϕt , 10^{22} n/cm ² (E>0.1 MeV)	Temp. (°F)
<u>Silicon (Cont'd)</u>				
29	+.030	48-3	1.97	863-889
25	-.095	48-11	4.19	1316-1468
26	+.11	↓	↓	↓
27	+.096			
28	+.12			
29	+.11			
25	+.068	44-7	3.53	1073-1263
29	+.077	44-7	3.53	1073-1263
<u>Molybdenum</u>				
30	-.28	44-19	1.53	740-747
31	-.28	↓	↓	↓
32	-.12			
33	-.034			
34	-.018			
30	-1.20	49-15	3.07	792-882
34	-0.49	↓	↓	↓
30	-.010	44-2	1.53	866-878
31	-.101	↓	↓	↓
32	-.020			
33	-.038			
34	-.048			
30	+.038	44-12	4.11	1318-1448
31	+.049	↓	↓	↓
32	+.068			
33	+.080			
30	+.015	45-7	3.54	1064-1253
34	-1.02	45-7	3.54	1064-1253
<u>Boron</u>				
13	-.18	49-17	2.20	856-885
14	-.18	↓	↓	↓
15	-.18			
15	-.18			
15	+.082	49-10	4.24	1359-1512
15	+.082	↓	↓	↓
15	-.006			
<u>Manganese</u>				
16	-.43	44-18	1.85	731-748
17	-.17	↓	↓	↓
18	-.13			
19	-.18			
16	-1.07	48-15	3.12	747-767
19	-.39	↓	↓	↓
16	+.069	49-8	3.86	1076-1289
17	+.059	↓	↓	↓
18	+.080			
19	+.049			
16	-.137	44-3	1.85	830-855
17	-.049	↓	↓	↓
18	+.0013			
19	-.053			
16		45-11	4.19	1329-1472
19		↓	↓	↓
16	+.067	44-11	4.24	1331-1473
17	+.074	↓	↓	↓
18	+.098			
19	+.058			

TABLE HEDL-19 (Cont'd)

Alloy No.	% $\frac{\Delta\rho}{\rho}$	Pin-Level	ϕt , 10^{22} n/cm ² (E>0.1 MeV)	Temp. (°F)
<u>Copper</u>				
20	-.21	46-18	1.93	711-729
21	-.21	↓	↓	↓
22	-.15			
23	+.054			
24	+.054			
20	-.47	48-15	3.12	747-767
24	+.011	↓	↓	↓
20	-.064	46-3	1.93	851-877
22	+.002	↓	↓	↓
23	+.17			
20	+.082	46-11	4.12	1327-1477
22	+.091	↓	↓	↓
23	+.167			
24	+.31	44-7	3.53	1073-1263
20	+.082	↓	↓	↓
<u>Cobalt</u>				
35	-.041	46-19	1.63	709-716
37	-.008	↓	↓	↓
39	-.033			
35	-.72	49-15	3.07	792-882
39	-.34	↓	↓	↓
35	-.029	46-2	1.63	876-877
37	-.062	↓	↓	↓
39	-.008			
35	+.10	46-12	4.00	1338-1464
39	+.11	↓	↓	↓
35	+.057	45-7	3.54	1064-1253
39	+.005	↓	↓	↓
<u>Nickel</u>				
40	-.084	48-19	1.66	734-741
41	-.10	↓	↓	↓
42	-.021			
40	+.11	48-12	4.07	1336-1463
41	+.057	↓	↓	↓
42	+.078			
40	-.056	48-2	1.66	875-886
41	-.056	↓	↓	↓
42	-.026			
40	+.079	46-7	3.48	1073-1271
41	+.022	↓	↓	↓
42	+.035			
<u>Chromium</u>				
41	-.10	48-19	1.66	734-741
43	-.050	↓	↓	↓
44	-.098			
41	+.057	48-12	4.07	1336-1463
43	+.063	↓	↓	↓
44	+.043			
41	-.056	48-2	1.66	875-886
43	-.034	↓	↓	↓
41	+.022	46-7	3.48	1073-1271
43	+.009	↓	↓	↓
44	+.014

FIGURE HEDL-57. Neutron Irradiation Swelling Data For 316 SS

FIGURE HEDL-58. Swelling In Carbon-Modified SS

4. Carbon and Nitrogen Variations

Swelling data for the alloy series representing carbon-nitrogen variations are shown in Figures HEDL-58 and 59. In these figures, the fluence is noted alongside each data point. It can be seen that there is a general decrease in swelling with increasing carbon content. The swelling data are plotted as a function of carbon content in Figure HEDL-60, where it is seen that this decrease is more pronounced in going from $\sim 0.0\% \text{ C}$ to $\sim 0.04\text{--}0.05\% \text{ C}$ than from $\sim 0.04\text{--}0.05\% \text{ C}$ to $\sim 0.13\% \text{ C}$ for the two temperatures represented on the figure. From previous investigations^(2,3) we know that thermal densification is occurring concurrent with the irradiation-induced swelling, with the resulting net change as shown in the figures.

This densification is more readily observable in the swelling data for the cold worked (20% reduction of area by swaging) alloys, shown in Figure HEDL-61. In this figure, it is seen that, for a given carbon concentration, a maximum densification is reached at an irradiation temperature of 1100°F , with no further densification at higher temperatures. Since thermal densification is thought to be a result of carbide precipitation⁽⁴⁾, and the extent of precipitation would be limited by the carbon content, it is quite reasonable that such maximum densification should occur. It was shown in Reference 2 that the densification obtained upon aging ($1500^\circ\text{F}/100\text{h}/\text{WQ}$) the same carbon-modified alloys was linear with carbon content. This same linearity is observed for the irradiated specimens, upon plotting the maximum densification observed vs carbon content for the irradiated cold-worked alloys, as shown in Figure HEDL-62. The values obtained for densification, $\sim 0.06\%$ to $\sim 0.14\%$, are the same as those values obtained for aging all of the alloys (in the 20% CW condition) at $1200^\circ\text{F}/100\text{hr}/\text{WQ}$, with two exceptions. The exceptions are the high molybdenum alloy (Alloy 34), and the high copper alloys (Alloys 23 and 34). These alloys exhibited densification of around 0.25%.

5. Silicon Variations

Swelling in the silicon-variation alloy series as a function of temperature and weight percent silicon is shown in Figures HEDL-63 and 64, respectively. Swelling in these figures is normalized to fluence in an attempt to clarify the effect of composition. The most striking features of the figures is the reduction in swelling which occurs in going from essentially zero silicon up to $\sim 0.5\% \text{ Si}$. Most materials specifications are fairly unrestrictive in limits on silicon levels, usually specifying only a maximum amount of silicon which may be included in the steel (See, for example, the Metals Handbook, Vol. 1 specifications for 304 and 316 stainless steel which limit silicon to one weight percent maximum). This variation in Si could be a contribution to the variations in swelling observed in some of the early swelling data⁽⁵⁾. With the silicon allowed to vary from 0% to 1.0%, Figure 8 shows that a variation in swelling of a factor of five would be possible.

The microstructure of these silicon-modified alloys has been examined and perhaps the most significant aspect is that the high-temperature increase in volume change (Figure HEDL-64, 1070°F) is not due to void formation. It is suspected that some solid-state reaction is responsible for the volume increase, but the nature of the reaction is unknown at this time.

6. Phosphorus Variations

Phosphorus was found to have a quite pronounced effect on swelling. Shown in Figure HEDL-65 are the results for variations in phosphorus from 0.001 to 0.039 weight percent. A

FIGURE HEDL-59. Swelling in Carbon-Modified SS.

FIGURE HEDL-60. Volume Change in Carbon-Modified SS as a Function of Carbon Content--Fluence $\sim 2.64 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$).

FIGURE HEDL-61. Densification in 20% CW Carbon-Modified Steel.

FIGURE HEDL-62. Densification in 316 SS as a Function of Carbon Content.

significant decrease in swelling is observed at the high phosphorus level. This decrease in swelling may be due to the ability of phosphorus to pin dislocations and inhibit climb. This would allow the dislocations to act as more efficient recombination sites and reduce the defect supersaturation, consequently reducing the swelling. This mechanism is supported somewhat by the work of Allen⁽⁶⁾ who noted an increase in yield stress resulting from increasing the phosphorus content in a dilute solid solution with iron. This would indicate that the phosphorus is inhibiting dislocation motion, probably by acting as a climb barrier. While iron is body-centered cubic and 316 stainless steel is face-centered cubic, we would expect the same type of phenomenon to occur in the stainless steel. With the dislocations thus restrained, their effectiveness as recombination sites would be enhanced due to their increased residence time at a given location. Also, the work of Rowcliffe⁽⁷⁾ suggests that a strong vacancy-phosphorus-atom binding energy exists. This would prohibit the vacancies from migrating rapidly to sinks and would again increase their residence time in a given volume of the lattice, increasing the recombination probability.

7. Manganese Variations

The variations in irradiation-induced swelling associated with variations in manganese content are shown in Figure HEDL-66. Shown in the figure is the decrease in swelling resulting from increasing the manganese content from 0.01 w/o to ~1-2 w/o.

In the low temperature region, there is a minimum volume change at ~2.0 w/o Mn, consistent with the observation of lower swelling in 316 stainless steel than in 304 stainless steel.

8. Molybdenum and Cobalt Variations

Swelling variations resulting from variations in molybdenum and cobalt content are shown in Figures 11 and 12 respectively. Both show an initial swelling peak around 840°F. In addition to this, the alloy with ~5 w/o Mo shows increased swelling around the 1100 to 1200°F range.

Again, as with alloys mentioned previously, increasing the weight percent of the varying element tends to decrease the swelling. This is probably due to a trapping mechanism which holds a vacancy or interstitial long enough for an increased amount of recombination to occur, thus lowering the defect super-saturation. It is highly probable that the major influence being represented by these data is a shift in the incubation period, as discussed earlier.

9. Chromium-Nickel Variations

The results of variations in chromium and nickel on irradiation-induced swelling in SS are shown in Figures HEDL-69 and 70 respectively. Some trends can be seen in the figures, such as a minimum in swelling at 17 w/o chromium and a decrease in swelling obtained by increasing the nickel from 12 w/o to 14 w/o. However, upon closer inspection of the figures, it is seen that the entire range of data points, for alloys which indicated some swelling, can be contained within the range of $\sim 0.01 \leq \% \frac{\Delta V}{V} / (\phi t) \leq 0.06$. The data for these alloys are definitely within experimental uncertainty. We therefore conclude that within the compositional range (15-19 Cr, 10-14 Ni), no significant variation in swelling behavior is observed. This of course applies strictly to the low-fluence data only.

10. Copper, Sulphur, Nitrogen and Boron

Variations in copper, sulphur, nitrogen and boron, to those levels indicated in Table HEDL-18, show no significant effect on irradiation-induced swelling.

FIGURE HEDL-63. Swelling in Silicon-Mounted SS as a Function of Temperature.

FIGURE HEDL-64. Irradiation-induced Swelling in Silicon-Modified SS.

FIGURE HEDL-65. Swelling in Phosphorous-Modified SS at a Function of $2.2 - 2.6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$).

FIGURE HEDL-66. Swelling in Manganese-Modified SS.

FIGURE HEDL-67. Effect of Molybdenum Variations on Swelling in SS.

FIGURE HEDL 68. Effect of Cobalt Variations on Swelling in SS.

FIGURE HEDL-69. Effect of Chromium on Swelling in Solution-Treated SS.

Figure HEDL-70. Effect of Nickel Content on Swelling in Solution-Treated SS.

FIGURE HEDL-71. Swelling vs Fluence for Molybdenum-Modified 316 SS in the Temperature Range 730 to 760°F.

FIGURE HEDL-72. Variance in Initialization Fluence with Molybdenum Content for 316 SS.

11. Discussion

From the previous section, it is apparent that some alloying modifications can improve the swelling behavior of 316 SS. To the fluence ranges investigated, additions of silicon, carbon, molybdenum and phosphorus appear beneficial. As was suggested earlier, this reduction in observed swelling is due to some mechanism increasing the residence time of point defects in a given volume of the metal lattice. This would manifest itself in an increase of the fluence required for the onset of steady state swelling (ϕt_i). Thus, in principle, one should be able to characterize this fluence (ϕt_i) in terms of the composition of the alloy. At the fluences attained thus far, such an analysis would be of dubious benefit. However, analytical techniques have been developed and will be illustrated with use of the low-fluence data. Swelling data from the molybdenum-modified alloys and from the 316 base alloys are plotted in Figure HEDL-71 for the low temperature range. It appears that the major effect of alloy modifications is to change the incubation Fluence (ϕt_i) and that once swelling begins it proceeds at the same rate (adjusted for temperature) for minor alloying variations. Extrapolation of the linear swelling curve to zero swelling established the incubation fluence (ϕt_i) for each alloy. If this zero-swelling intercept is then plotted as a function of composition, as shown in Figure HEDL-72, the variation in (ϕt_i) with alloy modification can be obtained. When higher values of fluences are reached, and the indication of identical swelling rates is verified, a method such as this can be used to identify optimum alloy modifications.

As mentioned above, re-irradiation of the specimens to fluences $\sim 10^{23}$ n/cm² (E>0.1 MeV) will provide the desired clarification for optimization of alloy content. This will be accomplished by means of an irradiation experiment consisting of three B-7a type pins to be irradiated in EBR-II beginning in July of 1974.

The experiment will consist not only of re-irradiated density specimens, but also of bi-axially-pressurized tube specimens which will provide a determination of the effects of alloy variations on in-reactor creep.

12. References

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V. EFFECTS OF ALLOY VARIATIONS ON CORROSION BEHAVIOR
OF POTENTIAL CLADDING AND DUCT MATERIALS

J. F. Bates, W. F. Brehm/J. J. Laidler, G. L. Grandy

A. OBJECTIVE

The objective of this investigation is to study the effects of minor alloy variations on the corrosion behavior of 316 SS in flowing sodium.

The results of this study, coupled with other studies of this type, will allow an optimization of reference cladding material for future use in the Fast Flux Test Facility (FFTF).

B. ACCOMPLISHMENTS AND STATUS

1. Introduction

Corrosion resistance in flowing sodium and resistance to irradiation-induced swelling have long been primary requirements for material designed for cladding and duct applications in Liquid-Metal Cooled Fast Breeder Reactors (LMFBR's). Recently it was discovered that minor alloy modifications of 316 SS result in improved swelling resistance.⁽¹⁾

Several of the alloys, as modified initially for the swelling studies, were exposed in the Source Term Control Loop (STCL) at HEDL to determine what effect these minor alloy modifications will have on the corrosion behavior of the material. These alloys have now received 5,000 hours and 8,000 hours exposure to sodium at 600°C (1120°F). This report summarizes weight change results for these specimens and future exposures of additional alloys.

2. Experimental Details

The specimens used in the experiment were tubes approximately 1/2 inch long by 0.230 inch OD by 0.015 inch wall thickness. The specimens were weighed on a micro-balance having a sensitivity of 1 microgram, and placed on the specimen stringer of the STCL just downstream of the radioactive specimens used in the Radiation and Repair Engineering Program. The specimens were removed periodically, re-weighed, and replaced on the stringer.

The composition of the specimens is shown in Table HEDL-20. The Source Term Control Loop, in which the specimens were exposed, is a pumped, nonisothermal facility. The conditions under which the specimens were exposed is as follows:

Specimen Temperature	1120 ± 3°F (604°C)
Cold Leg Temperature	800 ± 5°F (427°C)
Flow Rate	2 gal/min
Velocity Past Specimens	22 ft/sec (6.7 m/sec)

Oxygen levels were controlled by changing the cold trap temperature, and were monitored continuously by electrolytic oxygen meters, which in turn were calibrated by exposure of a vanadium wire to loop sodium by the standard technique.⁽²⁾

The loop and the radioactive mass transport experiments being conducted as part of the Radiation and Repair Engineering Program have been described previously.⁽³⁾ The oxygen level was 2.5 ± 0.2 ppm except for the 1000 hour interval between 1000 and 2000 hours, when the oxygen level was 0.5 ± 0.05 ppm. The 2.5 ppm level is higher than will be expected in FFTF/

TABLE HEDL-20
SPECIMEN COMPOSITION AND WEIGHT LOSS HISTORY

Alloy No.	Weight Percent ^(a)					Weight Loss (Mg/cm ²) ^(b)					Equivalent mils/year ^(c)
	C	N	Si	Cr	Ni	100h	200h	300h	500h	800h	
1	0.003	0.007	0.40	16.95	12.01	0.288	0.609	0.799	1.311		0.115
3 ^(d)	0.047	0.050	0.39	16.99	12.05	0.299	0.637	0.844	1.298	1.859	0.102
5	0.128	0.111	0.43	17.00	12.10	0.283	0.639	0.777	1.284		0.113
25	0.046	0.052	<9.01	16.88	11.98	0.331	0.684	0.849	1.307	1.852	0.102
29	0.045	0.050	1.96	17.13	12.39	0.398	0.819	0.875	1.480	2.013	0.111
40	0.045	0.050	0.40	14.86	9.96	0.186	0.501	0.540	1.081		0.096
41	0.046	0.052	0.38	14.89	12.01	0.279	0.660	0.775	1.381		0.122
44	0.041	0.049	0.41	18.97	12.11	1.88	0.486	0.609	1.198		0.105

(a) All other alloy constituents (except iron) remain at the AISI 316 SS specification.

(b) Based on weight at time = 0.

(c) Based upon weight loss at longest exposure time. Not corrected for Cr, Ni, Mn, Si leaching beneath the surface. Average value for irradiated 316 SS exposed in the same loop for 6000 h at 2.5 ppm, corrected for leaching, is 0.086 mils per year

(d) Alloy No. 3 is of AISI 316 SS composition.

LMFBR, but was chosen for the Radiation and Repair Engineer (R&RE) program to determine the effect of oxygen level on radioactive material corrosion as part of the companion experiment in STCL.

3. Results

The results of the corrosion studies are shown in Figures HEDL-73 through 76 for variations in carbon and nitrogen, silicon, chromium and nickel respectively. Table HEDL-20 provides a summary of the weight loss data.

Figure HEDL-73 shows that simultaneous variations in carbon and nitrogen have virtually no effect on the weight loss of the specimens. The extent of carbon depletion is being investigated at this time by Auger electron spectroscopy. Figure HEDL-74 shows that an increase in silicon from <0.01 wt% to ~0.4 wt% has little effect on the weight loss; a further increase to ~2.0 wt% does, however, increase the weight loss. Increases in chromium and nickel are shown to have opposite effects; an increase in chromium (Figure HEDL-75) resulting in a decrease in corrosion rate (Figure HEDL-75), and in increase in nickel resulting in an increase in corrosion rate (Figure HEDL-76). Table HEDL-21 summarizes the difference in weight change data for the longest exposures obtained.

4. Discussion

The results shown in Tables HEDL-20 and 21 show that the corrosion rates of all of the alloys are, in fact, quite modest, and that none of the alloys exhibit unacceptable corrosion rates. There is special interest in the silicon addition because of the beneficial effect of silicon upon swelling.⁽¹⁾ There is the possibility that the silicon is removed by an oxygen reaction to form sodium silicate, particularly at grain boundary locations, hence the increased weight loss observed in the high-silicon specimens. The microscopic examinations underway will yield more information.

FIGURE HEDL-73. Effect of Carbon-Nitrogen Variations on the Corrosion Behavior of Stainless Steel in Flowing Sodium.

FIGURE HEDL-74. Effect of Silicon Variations on the Corrosion Behavior of Stainless Steel in Flowing Sodium.

FIGURE HEDL-75. Effect of Chromium Variations on the Corrosion Behavior of Stainless Steel in Flowing Sodium.

FIGURE HEDL-76. Effect of Nickel Variations on the Corrosion Behavior of Stainless Steel in Flowing Sodium.

TABLE HEDL-21
WEIGHT CHANGE DIFFERENCES - LONGEST EXPOSURE

Alloying Element	Change in Composition (wt%)	Percent Change in Weight Loss (a)
C and N	0.005 → 0.11 (both elements)	-2.1%
Cr	15-17 → 19	-13.3
Ni	10 → 12	+27.8
Si	0.01-0.39 → 1.96	+8.7

(a) Based on weight loss of alloy with the lowest elemental weight percent of any in the particular series.

An increase in corrosion rate with increasing nickel content (10 to 12 wt%) is an anticipated result, as nickel is an element preferentially leached from stainless steel during exposure to flowing sodium. Nickel is thought to be released by an atomic dissolution process, independent of oxygen activity. Hence, increasing nickel content increases the driving force for dissolution of nickel at the metal surface. Nickel-base alloys such as the Inconel series have been observed to suffer higher corrosion rates than stainless steel.^(4,5) However, the observed 27.8% difference in weight loss is much larger than any reported difference between corrosion of 304 (8% Ni) and 316 (12% Ni) stainless steel.

A decrease in corrosion with an increase in chromium (15 to 19 wt%) is also somewhat surprising, as chromium is also leached out during exposure. These results can be better understood in light of the work of Roy, et al.⁽⁶⁾ It was determined that the corrosion rate of stainless steel in flowing sodium is dependent on the flow rate of the sodium past the specimens at velocities less than 15 ft/sec, corrosion in this case occurring by the diffusion of solutes and/or corrosion products across a fluid boundary layer adjacent to the surface of the metal. Above 15 ft/sec the thickness of this boundary layer decreases sufficiently that a surface reaction can occur and become the rate controlling step in the corrosion process. At the experimental velocity of 22 ft/sec, it is possible that a high chromium content could partially passivate the surface and retard this surface corrosion reaction.

An extreme example of this passivation effect is shown by the work of Fleitman and Issacs,⁽⁷⁾ who exposed pure chromium to oxygen-contamination sodium containing ~110 ppm oxygen, and observed formation of a grayish-colored film on the surface which prohibited corrosion of the chromium.

The effect of total (radioactive and nonradioactive) Mn content upon ⁵⁴Mn release was studied at 2.5 ppm oxygen. It was anticipated that the lower total Mn content would reduce the driving force for all Mn to enter the sodium from the steel. As shown in Table HEDL-22, there was no effect on ⁵⁴Mn release for Mn content in the range <0.1 to 4%. The irradiated specimens with variations in Mn content are being tested to determine if the total (radioactive + non-radioactive) Mn content affects the tendency for the ⁵⁴Mn to be leached from the stainless steel. Results from 6000 hour tests at 0.5 ppm oxygen will be available at the next reporting period.

TABLE HEDL-22
 VARIATION OF ⁵⁴Mn RELEASE WITH Mn CONTENT - STCL-1
 1120°F(604°C) 2.5 ppm Oxygen, 3000 hr

Specimen	Nominal Mn Content	Total ⁵⁴ Mn Release Percent of Total Activity Present in Sample
5b-1 (316)	1.5	0.89
5b-6 (316)	1.5	0.80
16B	<0.1	0.70
17B	1	0.70
18B	2	0.78
19B	4	0.75

5. References

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VI. IRRADIATION DAMAGE ANALYSIS

H. H. Yoshikawa

A. LONG REPLACEMENT SEQUENCES IN DISPLACEMENT CASCADE MODELING

D. G. Doran/T. K. Bierlein

1. Objective

The broad objective of this work is to determine analytically the damaging potential of neutrons of different energies and of electrons and ions used to simulate neutron effects in structural materials, and to apply this knowledge to the correlation of irradiation effects data obtained in different irradiation environments. The specific objective of the work reported here is to establish a mechanistic basis for the differences in swelling rates of stainless steel irradiated with neutrons, nickel ions, protons, and electrons.

2. Accomplishments and Status

The computer modeling of displacement cascade production and annealing has suffered from inadequate treatment of long replacement sequences. Such sequences permit the formation of a self-interstitial so far removed from its vacancy as to preclude recombination. Frequent occurrence of such events could significantly decrease the extent of recombination in a cascade.

A procedure was outlined in the preceding quarterly⁽¹⁾ for estimating the effect of replacement sequences on cascade annealing. It consisted of running the cascade simulation code MARLOWE⁽²⁾ with abnormally low values of the displacement energy (5 eV was used), then reducing the excessive number of defects by the random application of a spherical annihilation region. The final number of defects was selected to be numerically equal to $\sim 10 T_{\text{DAM}}$ where T_{DAM} (in keV) is that portion of the primary knock-on atom energy that is dissipated in atomic kinetic energy. The processed cascades were then given a simulated anneal⁽³⁾ at nominally 500°C, using a 32-site annihilation region.

Results were given previously for 2.5 and 5.0 keV cascades.⁽¹⁾ These are included in Table HEDL-23, along with new results for 20 keV cascades. The extent of annihilation at 20 keV is slightly greater than at 2.5 and 5.0 keV--35% compared with 29 and 30%, respectively. However, as shown in Table HEDL-23, the increase in the number of residual defects, relative to earlier results obtained without permitting long replacement sequences, is greater at 20 keV than at the lower energies. This is a reflection of the abnormally low number of defects found at 20 keV in the earlier work.⁽³⁾

It is perhaps more significant that the number of mobile vacancies is almost unaffected at 20 keV by this treatment of replacement sequences--just as found for the 2.5 and 5.0 keV cases. Of course it follows that vacancy clustering is appreciably enhanced. In contrast, the number of mobile interstitials is very much increased, with a concomitant decrease in interstitial clustering.

The simulated annealing of the 20 keV cascades was taken one step further in which the vacancy clusters were permitted to (instantaneously) dissociate. Some additional annihilation events occurred resulting in almost complete elimination of immobile interstitial clusters--the mobile fraction of interstitials rose from 84 to 97%.

TABLE HEDL-23
EFFECT OF MODIFYING MARLOWE RUNS TO INCLUDE LONG REPLACEMENT SEQUENCES (LRS)^a

Cascade Energy (keV)	Damaging Energy (keV) ^b	$E_d (= E_q)$ (eV)	Recombination Radius (hlu) ^c	Average Number of LRS's	Total Number of Pairs		Number of Defects, Post-Anneal ^d	
					Pre-Anneal	Post-Anneal ^d	Vacancies	Interstitials
2.5	2.23	5	5.23	6.9	24	17	6.0	15
		25	-	0	39	10	5.8	9
5.0	4.36	5	5.31	12.9	44	31	7.5	26
		25	-	0	77	18	7.0	14
20.0	16.4	5	5.05	46	153	99	19	83
		25	-	0	294	52	17	29

a. Values are averages of ten 2.5 keV, ten 5.0 keV, and five 20 keV cascades.

b. Based on the Firsov model as used in MARLOWE.

c. Average recombination radius (RR), in half-lattice units, applied prior to annealing. RR < 5.1 includes 248 lattice sites. RR > 5.1 includes 320 sites.

d. Recombination region consists of 32 sites, including 8 - 1st neighbors, 16 - 3rd neighbors, and 8 - 4th neighbors.

The values for T_{DAM} in Table HEDL-23 are for the Firsov model as used in MARLOWE. It is currently recommended,⁽⁴⁾ however, that the energy partition theory of Lindhard, et al be used to determine T_{DAM} in calculating displacement rates; the number of displacements is taken to be proportional to T_{DAM} . The Lindhard model values of T_{DAM} are given in Table HEDL-24, along with the numbers of residual defects per unit damaging energy. Included for comparison is the recommended values for the number of displacements per unit damaging energy for iron, nickel, and steels. The decrease in defect production efficiency with increasing energy is somewhat less pronounced than found earlier. Further decreases above 20 keV are not expected, based on earlier results.

TABLE HEDL-24
DEFECT PRODUCTION IN "STAINLESS STEEL" NORMALIZED TO
THE DAMAGING ENERGY OF LINDHARD ET AL

Cascade Energy (keV)	T_{DAM} (keV)	Displacements $\div T_{DAM}$	Total Pairs $\div T_{DAM}$	Mobile Vacancies $\div T_{DAM}$	Mobile Interstitials $\div T_{DAM}$
2.5	2.0	10.	8.5	3.0	7.5
5.0	3.75	10.	8.3	2.0	6.9
20.0	14.	10.	7.1	1.4	5.9

In summary, these estimates of the effects of long replacement sequences on the short-term annealing of MARLOWE displacement cascades suggest that:

- 1) Residual defect production in high energy cascades in stainless steel is not grossly over-estimated by the currently recommended procedure for calculating displacements. It is quite likely, however, that this procedure underestimates defect production at low energies such as are characteristic of proton irradiations.
- 2) The number of mobile vacancies is not significantly affected by inclusion of long replacement sequences. At a cascade energy of 20 keV (and presumably higher), the number of mobile vacancies is numerically equal to $\sim 1.4 T_{DAM}$ (keV).
- 3) Almost all interstitials surviving annihilation are mobile and escape from the cascade.
- 4) For long annealing times or high temperatures, complete vacancy cluster dissociation leads to only 10% additional annihilation in which the few remaining immobile interstitial clusters are converted to mobile defects.
- 5) Considerable work is needed to estimate defect production properly at energies below a few hundred eV.

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B. DAMAGE FUNCTION ANALYSIS OF CLADDING AND STRUCTURAL MATERIALS

R. L. Simons/T. K. Bierlein

1. Objective

Predictions of material performance in LMFBR applications must be made on the basis of tests in thermal and fast reactors not typical of LMFBR's. Damage functions which relate efficiency of a neutron in producing radiation damage to its energy must be defined for those property changes critical to design and safety analysis. Such damage functions are required to predict the damage effectiveness of irradiations in specific LMFBR service positions, as compared to test irradiations of cladding and structural materials.

C. THE PREDICTED EFFECTS OF EXPERIMENTAL AND MATERIAL PARAMETERS ON VOID NUCLEATION

R. W. Powell/J. J. Laidler

1. Objective

The objectives of this theoretical study are to evaluate the nature and relative magnitudes of effects that various experimental and material parameters have on the void nucleation process. This study, when coupled with other modeling efforts, should help to define alloy optimization techniques that will lead to the development of appropriate cladding and duct alloys for future LMFBR's.

2. Introduction

Modeling activities are a significant part of the Alloy Development Program and void nucleation is a subject receiving major emphasis. Preliminary work has begun in this area and indicates the value of further investigations.

The effects on void nucleation of various experimental and materials parameters were studied using equations previously developed for the coprecipitation of vacancies and interstitials into voids. (1,2) The steadystate nucleation rates and incubation times were evaluated from the following equations.

$$J_k = Z' \beta_k N_0 \exp (-\Delta G'_k / kT) \quad 1)$$

and

$$\tau = (2 Z'^2 \beta_k)^{-1} \quad 2)$$

$\Delta G'_k$ is the barrier to nucleation and is the maximum of the G'_n vs. n plot given by,

$$\begin{aligned} \frac{\Delta G'_n}{kT} = & \sum_{j=0}^{n-1} \ln \left\{ \frac{\beta_i}{\beta_v} + \exp \left[-kT \ln \left(\frac{C_v}{C_v^e} \right) \right. \right. \\ & \left. \left. + (4\pi)^{1/3} (3V)^{2/3} \sigma ((j+1)^{2/3} - j^{2/3}) \right] / kT \right\}. \quad 3) \end{aligned}$$

Similarly, Z' is the Zeldovich factor and is equal to the reciprocal of the width of this plot at kT units below $\Delta G'_k$. β_k is the rate that vacancies impinge on the void of critical size, n'_k , and is given by the relationship

$$\beta_k = 4\pi \frac{D_v}{a} C_v \left[\left(\frac{3}{16\pi} n'_k \right)^{2/3} + 2 \left(\frac{3}{16\pi} n'_k \right)^{1/3} + 1 \right], \quad 4)$$

where D_v is the vacancy diffusivity and C_v is the vacancy concentration. V is the atomic volume, σ is the surface energy, kT has its usual meaning and β_i/β_v is the ratio of interstitial to vacancy impingement rates at a void. β_i/β_v is taken as independent of void size and is typically set equal to 0.9 or 0.99, reflecting the slight preferential attraction of interstitials to dislocations.

In order to evaluate Equations 1 and 2 as a function of temperature, the vacancy supersaturation, C_v/C_v^e , must be calculated. For most temperatures of interest, vacancies have sufficient mobility that steady-state can be achieved in a short time, and therefore, the steady-state vacancy concentration given by Wiedersich⁽³⁾ is appropriate. Thus,

$$\frac{C_v}{C_v^e} = 1 + \frac{b}{2 C_v^e} \left[\left(1 + \frac{4\dot{n}}{v_v a b} \right)^{1/2} - 1 \right] \quad 5)$$

where

$$b = C_v^e + \frac{p}{a}$$

and $\dot{n}$ is the defect production rate, v_v is the vacancy jump frequency, a is a recombination factor and p is a sink probability term.

The computational procedure was to evaluate Equation 5 at a given temperature, then input the result into Equation 3, together with an estimate of $\beta_i \beta_v$, to obtain ΔG_k , n_k and Z' . Equation 4 was then evaluated and the necessary parameters were then inserted into Equations 1 and 2 to obtain the steady-state nucleation rate and incubation time for that temperature.

The effect that these two terms, J_k and τ , have on the overall process of swelling can be understood by considering Figure HEDL-77. If a bi-linear swelling law is assumed, the swelling at a given fluence is determined by both the steady-state void growth rate and by the fluence required to achieve steady-state growth (termed the incubation period*). The steady-state growth rate is controlled by diffusional kinetics, which will be incorporated into subsequent theoretical models. The incubation period can be treated with the present model since it consists of the true incubation time, τ , plus a growth stage prior to the time at which the spheres of influence of the voids begin to interact.⁽⁴⁾ This time-to-interaction should be a function of the mean spacing between voids, and therefore, be influenced by the nucleation rate. Thus, the calculated incubation time and the calculated nucleation rate should both affect the incubation period, but by different mechanisms.

3. Accomplishments and Status

Studies have been made of the effects of variations in material properties and experimental conditions on nucleation parameters. Variables investigated include surface energy, bias term (beta ratio, β_i/β_v), vacancy migration energy, lattice parameter, sink density and defect production rate. The dependence of nucleation rate and incubation time on these variables is illustrated in Figures HEDL-78 through 83. Figure HEDL-78 shows that an increase in surface energy will greatly reduce the nucleation rate, but will not affect the incubation time. On the other hand, as seen in Figure HEDL-79, as the bias term approaches unity (for example, if dislocation climb is restricted), the incubation time increases by orders of magnitude at all temperatures, while the nucleation rate is only slightly decreased.

*Note the distinction between the calculated incubation time, τ , and the observed incubation period.

Figure HEDL-77. The effect of variations in void formation kinetics on radiation-induced metal swelling.

The effect of the vacancy migration energy is somewhat more complex, as shown in Figure HEDL-80. It is evident that an increase in the migration energy (a decrease in mobility) will produce higher nucleation rates for most temperatures of interest. The predicted effect is very large, amounting to an increase of five orders of magnitude in the nucleation rate at 450°C, more than enough to compensate for the corresponding lower growth rate. The effect on the incubation time is also significant, with a larger migration energy producing a longer incubation time at low temperatures but a shorter incubation time at high temperatures. Thus, an increase in the migration energy results in a shift of the swelling curve to higher temperatures.

As indicated by Figures HEDL-83 & 84, the model used here predicts that reasonable changes in the lattice parameter produce essentially no effect on either the nucleation rate or the incubation time.

Figure HEDL-85 and 86 illustrate the effect of sink density on the nucleation parameters. A sink probability of 10^{-6} corresponds to a low sink density, while a probability of 10^{-4} is representative of a CW material containing about 10^{11} dislocations/cm². A heavily CW material would have a sink probability of about 10^{-3} . It should be noted that these sink probabilities are for stable sink densities which do not change during the irradiation. The predicted effect is very significant for the high sink density, which produces both a large

HEDL-99

FIGURE HEDL-78. Computer-generated curves that indicate how the surface energy should affect void nucleation. Damage rate is typical of the HVEM.

FIGURE HEDL-79. Computer-generated curves that indicate how the surface energy should affect void nucleation. Damage rate is typical of the HVEM.

FIGURE HEDL-80. The effect of changes in the relative impingement rates of interstitials and vacancies at voids on the nucleation process.

FIGURE HEDL-81. The effect of changes in the relative impingement rates of interstitials and vacancies at voids on the nucleation process.

FIG HEDL-82. The effect that changes in the Vacancy Migration Energy should have on Void Nucleation

HEDL-102

FIGURE HEDL-83. Typical changes in the lattice parameter should have no effect on the nucleation of voids.

FIGURE HEDL-84. Typical changes in the lattice parameter should have no effect on the nucleation of voids.

HEDL-103

FIGURE HEDL-85. Theoretical predictions of the importance of a stable sink density in the nucleation process. The sink density varies from about 10^9 dislocations/cm² (sink probability of 10^{-6}) to approximately 10^{12} dislocations/cm² (sink probability of 10^{-3}). Damage rate is typical of the HVEM.

FIGURE HEDL-86. Theoretical predictions of the importance of a stable sink density in the nucleation process. The sink density varies from about 10^9 dislocations/cm² (sink probability of 10^{-6}) to approximately 10^{12} dislocations/cm² (sink probability of 10^{-3}). Damage rate is typical of the HVEM.

HEDL-104

FIGURE HEDL-87. The expected effect on nucleation of changes in the usual HVEM damage rate of 1×10^{-3} dpa/sec.

FIGURE HEDL-88. The expected effect on nucleation of changes in the usual HVEM damage rate of 1×10^{-3} dpa/sec.

decrease in the nucleation rate and an increase in the incubation time. These two effects would combine to produce a significant increase in the incubation period.

The defect production rate can be quite easily varied during simulation experiments; Figures HEDL-87 & 88 show the effect of this parameter for displacement rates within the range of the HVEM. A decrease in the defect production rate by a factor of two should produce a lower nucleation rate and longer incubation time, but the effect is not large. However, an increase in the defect production rate by a factor of five does produce a rather large increase in the nucleation rate and incubation time with the observed effect increasing with temperature.

The importance of temperature can be observed in all of the preceding figures, where it is apparent that, beyond a given temperature range, the nucleation rate drops off dramatically with small increases in temperature. As further illustrated in Figure HEDL-89, an increase in the temperature from 560°C to 580°C can result in a substantial decrease in the nucleation rate (and therefore, the void number density). This rapid variation in the nucleation rate with temperature is supported by recent HEDL electron irradiation experiments with the HVEM. It was observed that the void number density in 316 SS decreased by an order of magnitude on going from an irradiation temperature of 560°C to 580°C.

FIGURE HEDL-89. An illustration of the rapid variation in void nucleation rate with temperature for conditions typical of an HVEM irradiation.

4. Conclusions

Current theory of homogeneous nucleation of voids in a supersaturation of vacancies and interstitials enables prediction of the effect of various experimental and material parameters on such observable quantities as the void number density and the incubation period. It was found that both large, stable sink densities and decreased defect production rates increase the incubation period by decreasing the nucleation rate and increasing the incubation time. The surface energy and the bias term are also expected to have a large effect on the incubation period, with the surface energy affecting the nucleation rate and the bias term influencing the incubation time. The vacancy migration energy primarily affects the nucleation rate, resulting in a shift of the swelling to higher temperatures if the migration energy is increased. Finally, the lattice parameter has essentially no effect on either the nucleation rate or the incubation time, and therefore, will not influence the observed void number density or the incubation period.

5. Expected Achievements

Work will be continuing on void nucleation studies as well as in other modeling efforts. It is intended to combine void growth with this void nucleation model in order to directly calculate the incubation period and the steady-state growth rate. In addition, it is planned to incorporate a dislocation loop nucleation theory⁽⁵⁾ into this model to study the relationship between loop structure and void formation. Subsequently, the void nucleation model will be modified to account for the effect of various types of impurities. The formulation of void nucleation and growth models will lead to their eventual use in alloy design for optimum materials performance in duct and cladding applications.

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D. ELECTRON BEAM CHARACTERIZATION FOR THE JEM-1000-4 HIGH VOLTAGE ELECTRON MICROSCOPE

D. S. Gelles, L. E. Thomas/J. J. Laidler

1. Objective

Of the various charged-particle irradiation techniques presently used to simulate high-fluence neutron irradiation effects in potential FTR alloys, electron irradiation in the 1 MV electron microscope offers a unique capability for observing the rapid accumulation of damage in controlled specimen regions sustaining up to 25 dpa/hr. Precise definition of the experimental conditions is required, however, before the results can be correlated with those of other irradiation methods. As a preliminary to extensive High Voltage Electron Microscope (HVEM) irradiations aimed at developing advanced low-swelling alloys for LMFBR fuel cladding, our irradiation practices are undergoing careful review. The objective of

this work is to describe electron beam profiles and fluxes and local temperature rises produced by these beams for the JEM-1000-4 electron microscope at HEDL.

2. Introduction

Use of the JEM-1000-4 high voltage electron microscope for irradiation simulation studies requires accurate determination of the electron flux distribution and other irradiation parameters. The purpose of this work is to provide an accurate description of the electron beam during operation under irradiation conditions. Information on local temperature rises will be reported later. This information is required for such future endeavors as: 1) electron flux determinations during electron irradiation; 2) optimization of electron irradiation conditions, so as to minimize beam heating while controlling point defect concentration gradients; and 3) mathematical modeling for analysis and interpretation of simulation results.

3. Experimental Details

As presently equipped, the JEM-1000-4 provides a choice of three focused beam sizes and continuous control over the total beam current up to a maximum limited by physical damage to apertures and other internal microscope components. The focused spot size is controlled by the excitation of the first condenser lens (C_1), with the focus itself controlled by the second condenser (C_2) lens excitation. Figure HEDL-90 shows a typically elliptical spot produced at the intermediate C_1 setting. In addition, various condenser apertures can be introduced to reduce the beam divergence and thereby permit detailed observations during irradiation.

FIGURE HEDL-90. Medium Spot Size Beam Shape at 20 μ A Beam Current, all Apertures Removed. The direction of beam sweep is indicated by the line.

The beams used for irradiations were too intense for controlled photo-graphic recording. Beam profiles were therefore obtained by sweeping the beam across a defining aperture (the field-limiting aperture used for selected-area diffraction) and simultaneously recording on an x-y recorder the current collected by a Faraday cup located below the aperture and the deflection voltage used to sweep the beam. The effective diameter of the defining aperture, $0.25\text{ }\mu\text{m}$, was determined photographically by comparing its image with the superimposed image of a magnification standard (a 28,800 line/inch grating replica placed in the normal specimen position). Knowledge of this effective aperture area allowed electron flux calculations from the Faraday cup measurement. Similarly, the beam spot widths were measured from micro-graphs and used to scale the recorded beam profiles.

4. Results

The maximum intensities obtained for the various beam conditions are listed in Table HEDL-25. From this table, it can be noted that peak intensity does not vary greatly with spot size. Peak intensity is a strong function of accelerator (total) beam current and is decreased by a factor of two when a large condenser aperture is inserted.

TABLE HEDL-25. BEAM PARAMETERS

Spot Size	Condenser Aperture	Accelerator Beam Current, μA	Maximum Flux, $\text{e}^-/\text{cm}^2\text{-sec}$	a, μm
Large	None	20	1.70×10^{20}	2.65
Large	None	10	1.00×10^{20}	2.40
Large	None	4	0.55×10^{20}	2.25
Medium	None	20	1.38×10^{20}	1.75
Medium	None	10	1.09×10^{20}	1.50
Medium	None	4	0.64×10^{20}	1.45
Small	None	20	1.23×10^{20}	1.00
Large	1000 μm ϕ	20	0.71×10^{20}	2.35
Medium	1000 μm ϕ	20	0.71×10^{20}	1.55
Small	1000 μm ϕ	20	0.67×10^{20}	0.90

A typical beam shape is shown in Figure HEDL-90 obtained at 20 μA accelerator beam current, medium spot size, with all apertures removed. The direction of beam sweep used is indicated by the line. As quite symmetric beam profiles were obtained, only half-profiles of the beams are reproduced in subsequent figures. Calculations of displacement rates were made on the basis of a 40-barn primary displacement cross section.

Each beam profile has been fitted to a Gaussian curve of the form

$$I = I_0 \exp - r^2/a^2,$$

where I is the beam intensity for a distance r from the center of the beam. I_0 and a are adjustable parameters, I_0 representing the maximum beam intensity and a describing the beam width. The values chosen for a have been indicated on each figure for each profile and are listed in Table HEDL-25. From these values for I_0 and a , values for I have been calculated at given r and are indicated by crosses on each profile. Excellent fit is apparent,

indicating that the electron beam is Gaussian in shape and can be described accurately by the expression given above.

Figure HEDL-91 demonstrates that, as the beam spot size is decreased, the maximum intensity is decreased by less than 25%, but the beam width is decreased by 60%. Figure HEDL-92 shows that the introduction of a 1000- μm condenser aperture decreases the beam intensity by as much as 55%, and this results in equal maximum intensities with little change in beam width, relative to the condition with the aperture removed, for all three spot sizes.

Figures HEDL-93 and 94 describe the effect of decreasing accelerator beam current on large and medium spot sizes, respectively. In both cases, decreased beam current can be seen to decrease beam widths by less than 18%.

4. Conclusions

The beam profiles for the JEM-1000-4 have been measured and the following conclusions can be drawn:

- 1.) Little decrease in beam intensity (and therefore dose rate) results from decreasing beam spot size.
- 2.) Little decrease in beam width results as the beam current is decreased.
- 3.) Introduction of a 1000- μm condenser aperture decreases the beam by up to 55%, has little effect on beam diameter, and results in equal maximum intensities for all three beam spot sizes.
- 4.) Beam profiles can be accurately described by the Gaussian expression

$$I = I_0 \exp - (r^2/a^2)$$

where r is the distance from the beam center, I_0 is the maximum beam intensity and the parameter a describes the profile width.

E. COMPARATIVE ELECTRON IRRADIATIONS OF FTR FIRST CORE TUBING

F. A. Garner, B. Mastel/J. J. Laidler

1. Objective

The objective of this work is to simulate, by means of dynamic electron irradiation with the high voltage microscope, the behavior of 316 SS at neutron fluences in excess of 9×10^{22} n/cm², which is the present limit of available comparable experimental data on both CW and annealed 316 SS. The results of simulation experiments will be correlated with neutron irradiation data and, on the basis of this correlation, used for guidance in the development of stress free swelling design equations.

2. Scope of Work

The swelling behavior of 316 SS under neutron irradiation has been shown to be very sensitive to both composition and thermal-mechanical history. The use of cold-working has been the most successful technique for the suppression of swelling, but there is some concern that radiation-assisted recovery may occur and eventually restore the

FIGURE HEDL-91. Beam Profile Variation With Spot Size (20 μA accelerator current, condenser aperture removed)

HEDL-111

FIGURE HEDL-92. Effect of 1000 m Condenser Aperture on Beam Profiles (20 μA Total Beam Current).

FIGURE HEDL-93. Beam Profile Variation With Accelerator current; large spot size, condenser aperture removed.

FIGURE HEDL-94. Beam Profile Variation With Beam current; medium spot size, condenser aperture removed.

solution-annealed swelling behavior. The thermal stability of the unirradiated CW microstructure has been investigated for clues to the material behavior under radiation service. Paxton and Holmes⁽¹⁾ found that the N-lot reference tubing could recrystallize in less than 2000 hours at 1400°F. Later work by Paxton and Straalsund⁽²⁾ showed that the one-hour recrystallization temperature of 20% CW 316 SS was determined by minor compositional changes within the RDT Standard E 13-8 limits and could vary from 1520 to 1620°F. More recently⁽³⁾ evidence has been presented that shows the 1600°F recrystallization times for the various vendor heats of first core cladding varied substantially and were very sensitive to the heat treatment used prior to the final 20% reduction in area.

If the onset of swelling in cold-worked materials is governed by the stability of the hardened microstructure, then it may be expected that those heats which take longer to recover would also exhibit greater suppression of swelling. To investigate this possibility, an irradiation program was initiated to study the electron-induced swelling behavior of each heat. Since there are both compositional and thermal-mechanical history differences in the various heats, a separate irradiation program was conducted on specimens of heat 81615, wherein the composition was constant, but the last annealing temperature was varied from 1850 to 2050°F.

3. Experimental Procedure

Specimens were punched in a manner that precluded further deformation from each of the tube lots designated N, S, BB, X, CN-13, CN-17; the latter three will be used in fabricating FTR first core cladding. The resulting curved 3-mm discs were hand polished on both sides through 600 grit silicon carbide to produce a flat disk 3 or 4 mils thick. Irradiations were performed in the standard manner at two displacement rates: 19.3 and 6.4 dpa/hr. The base irradiation temperature was 550°C and the estimated beam heating was 30 and 10°C respectively. Irradiations were terminated prior to the onset of serious surface perturbations which were defined in an earlier report⁽⁴⁾.

4. Experimental Results

Previous irradiations⁽⁵⁾ performed on 20% CW 316 SS involved the use of material which was prototypic in composition only, having used N-lot material which was cold-rolled to 20% reduction of cross-sectional area. The current irradiations proceeded on fully prototypic material which, being cold-drawn, had a microstructure that exhibited a high density of deformation twins not found in the cold-rolled structure. A comparison is made of the two microstructures in Figures HEDL-95 and 96. Since the heating cartridge had been replaced and the simulation procedure had been further optimized after the irradiations of cold-rolled specimens, a cold-rolled N-lot specimen was irradiated as a control. Figure HEDL-97 shows a comparison of observed swelling in the cold-rolled and cold-drawn N-lot reference specimens. The steady-state swelling rate was similar in both cases and somewhat higher than that observed at 550°C in the previous cold-rolled irradiation series. The incubation period was somewhat longer for the cold-drawn material, and might lead to the conclusion that this material has a superior swelling resistance by virtue of a greater resistance to void nucleation. It soon became apparent however, that local variations of swelling arise in nominally identical specimens. This is illustrated in Figure HEDL-98 where three separate irradiations of a first-core heat designated, CN-17, produced nearly identical steady-state swelling rates after substantially different non-linear incubation periods. There were no discernible differences in the local microstructure to account for

HEDL-114

FIGURE HEDL-95. Microstructure of 20% Cold-Rolled 316 SS (N-10t) After 14 dpa at 450°C.

HEDL-115

FIGURE HEDL-96. Microsturcture of 20% Cold-Drawn 316 SS (N-lot).

FIGURE HEDL-97. Comparison of Swelling Behavior of Two N-lot Specimens Under HVEM Irradiation.

FIGURE HELD-98. Comparison of HVEM-induced Swelling Behavior of Three Different Specimens of CN-17 Lot of 20% Cold-Drawn 316 SS.

these variations, however. Figure HEDL-99A shows a selection of typical irradiation results for four different heats of 316 SS that are designated as first-core steel. Also included are results of irradiations on tubing lots N and S, which are vendor qualification tubing lots. The steady-state swelling rates were measured to $1.4 \pm 0.3\%/dpa$ at a temperature calculated to be 580°C . This agreement is considered quite good when one considers the uncertainties associated with dose and thickness determinations, as well as the difficulty of sampling void populations in such heavily deformed microstructures. Irradiation results shown in Figure HEDL-99B proceeded at one-third the usual rate and swelling rates were lower ($1.1 \pm 0.2\%/dpa$), reflecting a calculated 20°C decrease in beam heating. Consistent heat-to-heat variations in the duration of the void incubation period were not discernible in either data set due to the range of local swelling variations within each heat. A typical void network produced in these irradiations is shown in Figure HEDL-100.

FIGURE HEDL-99. Selected Swelling Results of HVEM Irradiation of 20% Cold-Worked 316 SS.

It should be noted that some irradiations in which low void densities were developed began to exhibit symptoms of surface perturbations when irradiated to fluences higher than that recorded in Figures HEDL-97, 98 and 99. Artificial saturation or reduction in swelling was frequently observed when voids grew to sizes that were inconsistent with the void size and foil thickness criteria postulated by Garner and Thomas⁽⁶⁾. Most irradiations were terminated prior to initiation of surface-related problems.

Although the swelling rate did not change greatly when the displacement rate was dropped to 6.4 dpa/hr, the void number density changed from the low 10^{14} cm^{-3} range observed at

HEDL-118

FIGURE HEDL-100. Voided Microstructure Developed in 20% Cold-Drawn 316 SS at 6.42 dpa and $\sim 580^{\circ}\text{C}$ (19.3 dpa/hr).

19.4 dpa/hr to the low 10^{15} cm^{-3} range, probably reflecting a steep dependence of void number density upon temperature in the 550-600°C range. Powell⁽⁷⁾ has shown that void nucleation at lower displacement rates should lead to a lower void density and that the temperature-dependence is rather steep in this range.

In most of the areas selected, there were regions where voids nucleated relatively quickly and other regions which resisted nucleation more vigorously. This led to a nucleation behavior different from that observed in the cold-rolled N-Iot irradiations. The variation in void size distribution with dose indicates that void nucleation was greatly prolonged, due to late nucleation in those regions which had the highest concentration of deformation twins. Although the overall trend of most areas was to eventually yield to void nucleation, occasionally, the local deformation was so great as to preclude void nucleation in some areas for the duration of the experiment.

3: Discussion

Laidler⁽⁵⁾ has recently applied the results of limited neutron data and cold-rolled HVEM swelling rate data to arrive at the prediction that peak swelling for FTR goal-fluence will be 6% or less, but that swelling for the LMFBR demonstration plant goal fluence could be on the order of 20% or more. The solution-annealed results, which have been previously published, indicate that cold-working by any means represents a substantial improvement in the swelling resistance. The potential for further swelling unlimited by saturation is indicated by the large values of swelling obtained in solution-annealed material. The irradiations of fully prototypic material reported here confirm the general predictions drawn from the cold-rolled data and indicate that the beneficial effect of cold-working is retained to goal fluences. The mean incubation periods observed in these electron irradiations are consistent with the current estimates of fluence equivalences of 7 to 9 dpa per 10^{23} n/cm^2 ($E > 0.1 \text{ MeV}$) and the fact that prototypic material has not exhibited measurable swelling at one-half of the FTR goal fluence.

Since all irradiated areas eventually develop the same swelling rate, the integrated swelling should also develop at the same rate, allowing us to make predictions for bulk material response to irradiation. We cannot easily determine the bulk-integrated incubation period by HVEM irradiation, however. Broad-beam simulation techniques such as proton and nickel ion irradiations alleviate the problem of limited sampled volume utilized in swelling correlation development. However, the problem of the sensitivity of the void nucleation process to temperature and displacement rate remains unresolved. Laidler⁽⁵⁾ surmounted this problem by employing incubation fluences derived from neutron-induced swelling data.

It is encouraging that the absence of deformation bands in the cold-worked specimens did not appreciably change the swelling rate. This allows swelling rates to be determined in less complicated microstructures, easing the burden of void counting encountered in areas that are highly deformed.

In both solution-annealed and cold-worked irradiations, the total swelling rate did not appear to be sensitive to the void number density, which changed an order of magnitude within 20-30°C. This probably explains why the local suppression and extended nucleation of voids in heavily twinned regions did not appreciably alter the swelling rate from that observed in cold-rolled irradiations.

4. Expected Accomplishments

Irradiation and analysis of first core tubing samples will continue, to verify these findings. Micrographs from specimens of the 81615 heat, designated the Fabrication Variables experiment, will be analyzed to determine whether swelling is sensitive to the temperature of the heat treatment that proceeds the final reduction.

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F. SURFACE EFFECTS IN IRRADIATION EXPERIMENTS

F. A. Garner, B. Mastel, G. L. Guthrie/J. J. Laidler

1. Objective

The objective of this work is to extend the applicability of the thin-foil electron irradiation procedure by providing an experimental and theoretical understanding of the influence of surface proximity on void growth. The results of this work will then lead to an improvement in the experimental and analytical techniques used to simulate neutron-induced swelling in fast reactor structural materials.

2. Accomplishments and Status

A previous report⁽¹⁾ noted that voids nucleated in HVEM-irradiated 316 SS (both CW and annealed) disappeared abruptly between observations. In all cases, the voids vanished as their surfaces grew and protruded through the denuded zone boundary. The voids disappeared at the full range of void sizes from both surfaces and their volume was not conserved by ripening or coalescence events.

The range of possible mechanisms for void loss has been investigated further. All volume-conservative processes have been ruled out after studying the details of the void densities, sizes and distributions. Nonconservative processes require the void to either dissolve in place or to migrate to or toward the surface. The vanishing voids could either dissolve in the denuded zone or be annihilated at the surface. Migration, of course, requires a driving force, which may arise from temperature gradients, gradients in the point defect concentrations and stress field interactions or gradients.

While there are radial temperature gradients (parallel to the foil surface) in an HVEM specimen, there should be no measurable axial gradients. Defect concentration gradients

which are known to exist would cause a void to migrate toward the center of the foil, if at all. It is also possible that the surface influence may move toward the center of the foil due to thermal etching of the surface or elevation of the temperature toward the end of the experiments. Stress gradients of course will develop across the foil in response to the accumulated swelling; in some cases, specimens have been observed to develop a dome-shaped appearance by buckling in response to these stresses. It is not expected however, that these stresses are maintained in such thin foils, due to concurrent irradiation creep which should relieve these stresses as they develop. Wolfer (*) has proposed that voids could migrate up stress gradients due to the modification of point defect flow patterns. The sign of the stress would not matter, only the magnitude of the gradient. Our previous work indicated that voids could also drift toward surfaces due to stress field interactions, commonly referred to as image forces.

Several experiments have been conducted since the last report to study the nature of surface perturbations in HVEM irradiations. Analysis of many recent HVEM irradiations shows that a variety of potentially misleading surface effects can occur. In the solution-annealed 304 SS specimen shown in Figure HEDL-101, an artificially low swelling rate was observed due to void loss at 450°C. Correction factors developed in a previous report for void intersection with the denuded zone were applied with some success to calculate an unperturbed void growth rate.

Under certain experimental conditions, the loss of voids can be so substantial that such correction factors cannot be applied. Figure HEDL-102 shows the swelling behavior of several CW 316 specimens which appeared to saturate or decline in swelling after 15 dpa. In one case, the saturation was due largely to a decrease in void concentration, while in the other case, the void concentration decrease was accompanied by a saturation of the mean void size, as shown in Figure HEDL-103.

To determine whether saturation of swelling can arise due to void interaction with the surfaces, an experiment was conducted wherein the only variable allowed to change was the foil thickness. Two irradiations of SA 316 were performed, with one foil at a thickness of 4.7 times the measured denuded zone thickness, and the other with a thickness of 2.7 denuded zone widths (1.2 and 0.68 μm). Figure HEDL-104 shows the swelling behavior for the two irradiations which proceeded at 530°C and 10 dpa/hr. Two small areas were chosen for analysis in the thinner foil, to avoid a bend contour that obscured the voids at various times during the irradiation. Although the swelling rate in both experiments was initially the same, the swelling rate in the thinner foil quickly leveled off, while the thicker foil continued to swell to high levels without saturation. Analysis of the void size histories recorded during the experiment with intentional surface perturbations, as shown in Figure HEDL-105 leads to the following conclusions. Void diameters are limited to a width that cannot far exceed the total thickness minus two denuded zone widths. Voids which coalesce late in life to yield diameters larger than this value vanish before the next observations. When voids disappear, subsequent nucleation events can repopulate the region,

* W. G. Wolfer, Private Communication

FIGURE HEDL-101. Correction of HVEM-Induced Swelling Data for Loss of Voids Due to Intersection of Void Surfaces with Denuded Zone Boundary.

FIGURE HEDL-102. Examples of Surface Perturbations in HVEM Irradiations Leading to Saturation or Reduction in Swelling with Dose.

FIGURE HEDL-103. Distribution of Void Sized and Densities for Specimens Shown in Figure HEDL-102.

leading to an oscillation of the swelling about some saturation value. In this atypical experiment, only those voids whose centers were roughly equidistant from both surfaces managed to survive and these saturated in size. All others eventually vanished, due to surface perturbations which may or may not be balanced from both surfaces.

Another experiment previously reported was analyzed further to determine whether the disappearance of voids could be related to buckling of the foil, leading to a preferential loss by extrusion of voids to one side of the foil. Figure HEDL-106 shows the void center positions plotted for a 500°C irradiation of 20% CW 316. Stereographic observation at 21.2 and 28.3 dpa revealed that many voids disappeared between observations. The center positions of voids that did not vanish are plotted as solid circles, while voids which disappeared are plotted as open circles. The stereomodel was divided roughly down the center parallel to the surface and the various categories of voids counted on both sides. On the upper surface 88 voids vanished, while the lower surface lost 132. However, the upper region contained only 258 total initial voids, while the lower region contained 404 voids. The loss ratios of 34.1 and 32.7% are considered statistically insignificant, leading to the conclusion that buckling and void extrusion is not responsible for the void loss. There was also no discernible loss pattern across the irradiated area. From this irradiation, we cannot determine whether coalescence events preceded each void loss, but analysis of previous experiments reported by Laidler and Mastel⁽²⁾ proved that coalescence events were not a necessary precursor of void loss.

FIGURE HEDL-104. Comparison of Results for Two HVEM Irradiation of SA 316 SS with Different thicknesses.

Another experiment was conducted to determine whether voids disappeared due to thermally-activated processes in the absence of irradiation, when the irradiation was interrupted for photographic retrieval of data. An irradiation of 20% CW 316 (BB-1ot) was conducted to 19.3 dpa at 550°C (19.3 dpa/hr). The heater was then maintained at temperature for 30 minutes with the beam current reduced to observation levels. The specimen was observed by remote video-camera and recorded on videotape. No change in the void population was observed, as confirmed by further photography. The specimen was then held at 600°C and 650°C for 30 minutes each with no discernible change in void sizes or number. The final void structure is shown in Figure HEDL-107. Note the one obvious void coalescence event which remained essentially unperturbed at these temperatures. The conclusion drawn here is that the processes which lead to void annihilation occur during irradiation and are not related to thermal annealing effects.

In the final experiment to be discussed here, some significant light was shed on the void disappearance process. This irradiation proceeded at a thinner foil thickness than normally employed at 550°C for 20% CW 316, the specimen being only about $0.70 \mu\text{m}$ thick. The resulting 2000 Å thick void-bearing zone quickly yielded a large fraction of vanishing voids at each successive observation. Whereas in previous irradiations, the voids were observed to disappear between observations, sufficient photographs were taken in this case to show that

FIGURE HEDL-105. Comparison of Void Size Histories for Two Irradiations Described in Figure HEDL-104.

FIGURE HEDL-106. Projection of Void Center Positions at 21.2 dpa in 500°C Electron Irradiation of 20% CW 316 SS.

GURE HEDL-107. Voided microstructure in 20% CW 316 obtained after 19.3 dpa at 550°C and annealing for one-half hour each at 550, 600 and 650°C; no discernible change in the microstructure was produced by annealing.

many of the voids first grew and later proceeded to shrink over the period of several observations, finally vanishing completely. Figure HEDL-108 and 109 show several micrographs where voids were observed in the process of shrinking. Superimposed on these voids are the outline of the void at the previous observation. Voids which disappeared completely are marked with crosses. These shrinking voids were interspersed with other voids which grew and occasionally nucleated in the same region. The actual location of the denuded zone boundary was difficult to determine due to the low density of voids, but it was obvious that the shrinking voids were close to the denuded boundary, but not close to either foil surface. The conclusion drawn here is that, although voids may migrate under surface-induced forces, they rapidly shrink in the denuded zone and need not intersect the foil surface in order to be annihilated.

In a previous report, we presented a method of calculating the transit time for a void to diffuse from a given position in the interior of a solid to a position where it touched the external free surface. This was done numerically, using work of Willis and Bullough⁽³⁾, together with that of Shewman⁽⁴⁾. In the previous report, a table was given showing transit time for a 500 Å void to traverse various distances to make contact with the surface of the solid. This work has been extended, and now provides a more useable description of the effect of the free surface. Results of the numerical integration procedure have been reduced to a simple formula which facilitates analytical treatment and provides better insight to the question.

In the Bullough paper⁽³⁾, the larger of the two cavities is given boundary conditions such that the surface of the cavity is stress-free. Thus, for large ratios of cavity radii and close separation distances, the smaller cavity is essentially in the presence of a planar free surface. However, Bullough only tabulated the interaction energy and not the force, and only for the case of a 10/1 radius ratio. We have taken his formula (an infinite series) for an arbitrary ratio of cavity radii and an arbitrary spacing and have devised means for calculating the truncation error for this series, which is poorly convergent at large ratios of radii and small cavity-to-cavity separation distances. This program was run for a wide variety of ratios of cavity radii, as well as for several ratios of λ/μ , the Lamé constants and a variety of surface-to-surface separation distances. It was found that in the case of ratios of radii and ratios of (large radius/separation distance) which simulated a single cavity near a flat surface, the force, F , is given by

$$F = 1.55 \frac{r^6}{c^4} \frac{(p - \frac{2\gamma}{r})^2}{\mu} \quad (1)$$

to an accuracy of 10% for ratios of λ/μ varying from 1.4 to 2.6. (A ratio of 2.0 gives a Poisson ratio of 0.33). In the formula above, r is the radius of the cavity, c is the distance from the center of the cavity to the free surface, μ is a Lamé constant = C_{44} , γ is the surface energy, and p is the gas pressure in the void. The factor 1.55 is dimensionless. The validity of the simple formula is independent of the values of the radii and separation distances, provided that the geometry simulates a single cavity near a flat free surface.

9.7 dpa

1.0 μm

14.4 dpa

FIGURE HEDL-108. Void Loss by Shrinkage in 20% CW 316 SS at 14.4 dpa. Void Sizes at Previous Observations are also Shown. Crosses Indicate Voids

14.4 dpa

1.0 μM

19.3 dpa

FIGURE HEDL-109. Void Loss by Shrinkage in 20% CW 316 SS at 19.3 dpa. Void Sizes at Previous Observations are also Shown. Crosses Indicate Voids that Completely Dissolved.

The formula has been integrated using Shewman's equation for drift rate of a void due to surface diffusion, with the result that

$$\frac{dx}{dt} = \frac{1.55}{c^4} (KT\pi\mu)^{-1} D_s \delta \Omega r^2 \left(P - \frac{2\gamma}{r}\right)^2 \quad (2)$$

in the equation above, K is Boltzmann's constant, T is absolute temperature, D_s is the surface diffusion constant, δ is the thickness of the diffusion layer, Ω is the atomic volume, r is the radius of the small void which simulates a void in a foil, dx/dt is the drift velocity toward the larger void or toward the surface of the foil, and the other terms have been defined previously. Recognizing that C and X represent the same quantity, and integrating, we obtain

$$(t - t_0) = KT\pi\mu [7.75 D_s \delta \Omega^2 (P - \frac{2\gamma}{r})^2]^{-1} (X^5 - X_0^5) \quad (3)$$

for the elapsed time associated with a change in position of the center of the cavity from a position a distance X from the void surface to one a distance X_0 from the void surface. When $X_0 = r$, we have a case where the cavity touches the surface. Therefore the time-to-touch is given by:

$$t_{\text{touch}} = KT\pi\mu [7.75 D_s r^2 \delta \Omega (P - \frac{2\gamma}{r})^2]^{-1} (X^5 - r^5) \quad (4)$$

This time has been calculated and compared with the results given by numerical differentiation and integration using Bullough's table in the previous report. The average discrepancy was 4% and the maximum was 8% between the two sets of results. In general, the transit times using the formula above are slightly smaller because the flat surface of an infinitely large cavity exerts a greater force on the small cavity than that given by Bullough's 10/1 radius ratio, which was used in the previous report. A number of conclusions can be drawn from examination of these results:

- 1) For zero gas pressure, two cavities whose centers are equal distances from the surface of a solid will drift at equal velocities even if their radii are different.
- 2) If gas is added to these same cavities to give equal values of $(P - \frac{2\gamma}{r})$, the larger cavity will drift faster than the small one.
- 3) When $(P - \frac{2\gamma}{r}) = 0$, there is no drift velocity for cavity migration to the boundary which is imparted by surface stresses.
- 4) If one observes the leading edges instead of the centers of the cavities, two gas-free cavities with equal (cavity surface) to (foil surface) distances will exhibit drift velocities with the smaller cavity moving faster, (because its center is closer to the foil surface and Eq. (2) involves center to surface distances).

3. Conclusion

Void loss events are due to rapid shrinkage under irradiation and are not due to thermal annealing. Surface-induced migration may be occurring, but intersection of the void with the surface does not occur. This requires that the net point defect flux or "growth fluid" be

negative just beyond the denuded zone. This is in agreement with previous theoretical studies⁽¹⁾. Void losses have been observed over a temperature range of 450 to 650°C for solution-annealed 304, 316 and 316L SS as well as 20% CW 316 SS. Experiments are now routinely conducted in a manner to minimize surface perturbations and experiments are terminated prior to the occurrence of surface-dominated behavior. Theoretical analysis confirms the possibility of void motion and indicates that radiation-induced point defects are necessary to provide the surface diffusivity required.

4. Expected Achievements

Future experimental studies are planned to determine the nature of void loss mechanisms so as to reduce the extent of surface perturbations in HVEM experiments. The use of lower irradiation rates and neutron preconditioning of specimens have already shown some promise. Analytical studies are currently in progress to predict the temperature and microstructural dependence of denuded zones. Void migration kinetics are also being studied to explain void loss mechanisms.

5. References

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Naval Research Laboratory

I. ADVANCED REACTOR STRUCTURAL MATERIALS

A. AEC-CE-NRL COOPERATIVE PROGRAM ON IMPROVED (RADIATION RESISTANT) A533-B STEEL PLATES AND WELDMENTS

J. R. Hawthorne

A cooperative AEC-CE-NRL research program is exploring trends in radiation effects for commercially produced ASTM A533-B steel specially tailored for improved resistance to radiation embrittlement. The program involves plates, weld deposits, and weld heat affected zones (HAZ) and is directed primarily to the improvement of radiation resistance at 550°F (288°C) with progressive reductions in allowable copper content. Overall objectives are: 1) to develop enough information to formulate specifications for steels for nuclear service; and 2) to delineate associated embrittlement trends for vessel design and operation.

Three series of materials were selected for study that represent differing levels of copper content control: Series 1 - normal copper content ($\geq .15\%$ Cu) typical of nonimproved commercial steel production (pre-1971); Series 2 - low copper content (0.10% Cu max with 0.012% P max), representing improved steel production; and Series 3 - extra low copper content (0.06% Cu max) considered the practical lower limit for copper content control. In the case of Series 2, the copper and phosphorus content limits were chosen to match limits being proposed for supplemental ASTM A533-B steel specifications for nuclear service. Assessments of the radiation resistance of Series 1 and 2 materials have been made and are summarized below; assessments of the radiation response of Series 3 materials are still in progress. A more detailed report on the investigation of Series 1 and 2 materials is available.⁽¹⁾

The experimental plan centered on evaluations of Charpy-V (C_v) notch ductility before and after 550°F (288°C) irradiation to two fluence levels: 2 to 3×10^{19} and 5×10^{19} n/cm² >1 MeV. Resistance to radiation exposure was to be judged both in terms of the increase in C_v transition temperature indexed to the C_v 30 and C_v 50 ft-lb levels, and in terms of the decrease in C_v upper shelf energy level. Determinations of strength elevations accompanying notch ductility changes (higher fluence exposure only) were an additional part of the plan. Finally, notch ductility recovery with post-irradiation 650°F (343°C)-168 hour heat treatment was also to be explored. Except for the evaluation of strength changes, post-irradiation assessments of Series 1 and 2 materials are complete. Chemical compositions of the materials are given in Table NRL-1.

Several observations concerning material performance as a function of copper content were possible from the irradiation data. The most important of these is illustrated in Figure NRL-1 where C_v 30 ft-lb transition temperature increases determined in this study are compared to trends previously observed for A533-B steel (plates, welds) representing noncontrolled steel production and for the ASTM A302-B reference plate.^(2,3) Closed symbols depict data for Series 1 materials; large open symbols represent data for Series 2 materials. Small open symbols are

TABLE NRL-1
CHEMICAL COMPOSITION OF A533-B PLATES AND WELD DEPOSITS
CHEMICAL COMPOSITION (wt%)

Material	Cu	P	C	Mn	S	Si	Ni	Cr	Mo
Normal Copper, Series 1									
Plate 1	.17	.009	.23	1.29	.015	.21	.56	.10	.57
Plate 2	.24	.008	.25	1.40	.011	.23	.62	.11	.59
Weld 1	.36	.015	.14	1.38	.012	.22	.78	.07	.55
Weld 2	.20	.016	.13	1.11	.013	.17	.04	.05	.53
Low Copper, Series 2									
Plate 3	.09	.009	.20	1.29	.017	.22	.58	b	.57
Plate 4	.09	.011	.21	1.38	.018	.28	.66	b	.52
Weld 3	.07	.010	.15	1.15	.010	.25	.12	.04	.59
Weld 4	.05	.004	.15	1.25	.010	.19	.09	.06	.62
Normal Copper, Series 1		AI	N	V	As	Sn	Other		
Plate 1		.027	.007	.001	<.01	.003	a		
Plate 2		.02	.007	.002	<.01	.008	a		
Weld 1		.004	.012	.003	<.01	.001	a		
Weld 2		<.001	.008	.01	.013	.004	a		
Low Copper, Series 2									
Plate 3		b	b	b	b	b	---		
Plate 4		b	b	b	b	b	---		
Weld 3		.006	.005	.008	.010	.003	a		
Weld 4		.001	.004	.007	.006	.002	a		

^aCo <.013; Nb, Ti, W≤.01; Zr≤.002; B≤.0008

^bnot determined

data for the A533-B steel plate and weld deposit (0.03% and 0.05% Cu respectively) fabricated in forerunner commercial-scale demonstration tests of improved radiation resistance.^(4,5) The new data are seen to form two distinct patterns from which a benefit of low copper content to radiation resistance is clearly evident. Note further that the transition temperature increases for Series 2 materials do not exceed 100°F (56°C) for fluences up to 5×10^{19} n/cm². Accordingly, the limitations on copper content for nuclear service proposed by ASTM for A533-B plate (0.10% Cu max) and by AWS for A533-B weld deposits (0.08% Cu max) appear both justified and sufficiently low for the anticipated fluence applications.

FIGURE NRL-1. Summary of Charpy-V 30 ft-lb Transition Temperature Changes with Irradiation Observed for Series 1 Materials (filled symbols) and Series 2 Materials (large open symbols). Earlier determinations for low copper content A533-B materials from commercial scale demonstration tests are also given (small open symbols). A benefit to radiation embrittlement resistance by a low copper content (0.10% Cu max) is clearly evident. Performance trends reported for nonimproved A533-B plates and welds and for the ASTM A302-B reference plate are indicated for comparison.

The data also indicate comparable radiation embrittlement trends for Series 2 materials and demonstration test materials. Both plates from Series 2 had a copper content of 0.09% Cu, whereas plates from the 30-ton demonstration melt had a copper content of 0.03% Cu. Accordingly, it is speculated that the low copper content Series 2 plates and the extra low copper content Series 3 plates will be found to perform equally well when results for the latter are available. That is, a further reduction in allowable copper content from 0.10% Cu max (ASTM specification) to 0.06% Cu max (best steelmaking practice) may result in only a small improvement to radiation resistance for A533-B steel for the fluence levels investigated here (5×10^{19} n/cm²).

Within the trend band for low copper content materials, a difference in weld deposit versus plate performance is suggested which may be a reflection of the somewhat lower copper and phosphorus contents of the weld deposits. Weld 4 with the lowest copper content and phosphorus content of the series also exhibited the best radiation resistance. The mixed

performance of HAZ relative to parent plate is not understood. The results suggest that current proposals for the elimination of HAZ specimens from reactor surveillance programs may be premature where Series 2 type of materials are involved. It is pointed out, however, that although larger increases in transition temperature were recorded for the Series 2 HAZ, their absolute transition temperatures did not exceed those of the parent plate after irradiation because of lower starting points.

Table NRL-2 summarized pre-post-irradiation upper shelf energy determinations for the materials. The upper shelf energy values after high fluence irradiation were found to be greater than 80 ft-lb in all cases except for Weld 1. Here, the high copper content of Weld 1 (0.36% Cu) is believed partially responsible and is noted to be above the general range for noncontrolled commercial production weldments. The remaining Series 1 materials demonstrate well the benefit of a high initial upper shelf energy. To help assure adequate post-irradiation upper shelf levels, ASTM and AWS proposed specifications for nuclear grade A533-B include restrictions on sulfur content (plate: 0.015% max; weld deposit: 0.013% max) as well as on copper and phosphorus content. The results of this study suggest that the proposed sulfur content restrictions are adequate for anticipated fluence applications.

TABLE NRL-2
POST-IRRADIATION CHARPY-V UPPER SHELF ENERGY LEVELS
OF THE A533-B PLATES, WELD DEPOSITS AND WELD HEAT AFFECTED ZONES (HAZ)

Material	Fluence ($\times 10^{19} \text{ n/cm}^2 > 1 \text{ MeV}$)		C_v Upper Shelf	
	ϕ_{CS}	ϕ_{FS}	Energy (ft-lb)	Δ (ft-lb)
Plate 1	1.8	2.1	93	22
	5.9	6.7	94	21
HAZ 1	2.5*	2.9*	114	30
	5.9	6.7	~114	~30
Plate 2	3.2	3.7	84	~26
	5.3	6.1	86	~24
Weld 1	3.0	3.4	56	51
	5.3	6.1	56	51
Weld 2	3.0	3.4	~98	~31
	5.3	6.1	~98	~31
Plate 3	3.6	4.2	107	(+) 3
	5.5	6.3	111	(+) 7
HAZ 3	3.6	4.2	122	11
	4.9*	5.7*	~122	~11
Plate 4	2.2	2.5	130	(+) 11
	4.3	5.0	130	(+) 11
HAZ 4	2.2	2.5	158	(+) 25
Weld 3	4.1	4.6	155	~0
	5.0	5.7	155	~0
Weld 4	4.1	4.6	147	~0
	5.0	5.7	154	(+) 10

* average fluence; actual values varied between two groups of specimens.

Primary conclusions drawn from the experimental results of the program to date have been summarized⁽¹⁾ as follows:

- 1) A major reduction in radiation sensitivity is achieved in commercial production plates and weld deposits with reduced copper content (0.10% Cu max) compared to a normal copper content ($>0.15\%$ Cu). The reduction in radiation sensitivity is evident as a smaller transition temperature increase and as a smaller upper shelf energy decrease with irradiation.
- 2) Proposed ASTM and AWS specifications placing restrictions on copper, phosphorus, and sulfur contents for nuclear grades A533-B plates and weld deposits appear justified and well formulated for nuclear service applications.
- 3) Low copper content materials of this investigation compare well in performance with low copper content materials from earlier demonstration tests. It is speculated that extra low copper content materials (0.06% Cu max) will exhibit only a small additional increase in radiation embrittlement resistance at moderate fluence levels over the performance of low copper content materials (0.10% Cu max).
- 4) Low copper content weld metals tend to show higher radiation resistance than low copper content plates. The performance of weld HAZ relative to parent plate appears mixed; however, the notch ductility of the HAZ after high fluence exposure was found equal to, if not better than, the notch ductility of parent plates.
- 5) Post-irradiation 650°F (343°C)-168 hour heat treatment for notch ductility recovery is most effective in normal copper materials where upper shelf and transition temperature shifts appear greatest after irradiation. For the normal copper content materials investigated, upper shelf levels after post-irradiation heat treatment were 76 to 116 ft-lb as a result of 40% or more recovery. Recovery in transition temperature ranged from 25 to 100°F or 25 to 35%. Within the range of fluence investigated, notch ductility recovery was not appreciably governed by exposure level.

B. STRENGTH AND NOTCH DUCTILITY OF SELECTED STRUCTURAL ALLOYS AFTER HIGH FLUENCE, 550°F (288°C) IRRADIATION

J. R. Hawthorne and H. E. Watson

Tensile strength and C_v notch ductility changes with elevated temperature, high fluence irradiation have been explored for several low alloy structural steels. The study was addressed primarily to metal fracture resistance at upper shelf temperatures and encompassed A302-B, A533-B, A543-1, 9Ni-4Co-20C, 12-6PH, and 12Ni-5Cr-3Mo steel compositions. Material forms included plate, forging, and weld deposit. Material types, compositions, and heat treatment conditions are indicated in Tables NRL-3 and 4. Specimen irradiations were conducted in the Advanced Test Reactor (ATR) in a water-cooled loop. The nominal irradiation temperature was 550°F (288°C); the neutron fluences ranged from 1 to 7×10^{20} n/cm² >1 MeV. Specimens of one A543-1 plate were also irradiated at 600°F (316°C). Irradiation times for 550°F (288°C) exposures were either 360 or 1095 hours; the irradiation time for the 600°F (316°C) exposure was 1771 hours. A full description of the investigation and of the experimental results are given in Reference 6.

Primary observations on material yield strength trends are illustrated in Figures NRL-2 through 4. Radiation-induced changes in tensile strength paralleled, but were less than, yield strength changes. Several performance patterns are apparent from the data. First, there is no evidence of a saturation of radiation effect (A302-B, A533-B, and A543-1 material) where results are available for more than one fluence. Second, a linear relationship between yield strength and temperature is found for both irradiated and unirradiated material conditions for the range

TABLE NRL-3 - CHEMICAL COMPOSITION OF PLATES, FORGINGS AND WELD DEPOSITS

Material Identification		Composition (wt %)									
Material Type	Thick-ness (in.)	C	Mn	P	S	Si	Ni	Cr	Mo	Cu	Other
<u>Plate</u>											
A302-B (ASTM Reference)	6	0.23	1.35	0.015	0.021	0.22	0.22	0.12	0.52	0.22	0.01 V, 0.038 Al
A533-B (Demo. Melt)	6	0.17	1.22	0.008	0.008	0.19	0.58	0.06	0.50	0.03	0.02 V, 0.015 Al, 0.02 Sn
A543-1	8	0.17	0.32	0.011	0.016	0.25	3.37	1.92	0.50	0.05	0.02 V, 0.02 Al
A543-1	6	0.18	0.30	0.006	0.019	0.25	3.25	1.71	0.52	0.20	0.02 V
9Ni-4Co-.20C	2	0.18	0.20	0.006	0.009	0.02	9.00	0.77	0.95	0.10	0.08 V, 4.50 Co
<u>Forging</u>											
12-6 PH ^a (H1025)	4	0.038	0.01	0.010	0.007	0.06	5.96	11.82	...	3.01	0.13 Nb, 0.007 N
12-6 PH ^a (H1100)	4	0.038	0.01	0.010	0.007	0.06	5.96	11.82	...	3.01	0.13 Mb, 0.007 N
<u>Weld Deposits</u>											
A533-B-2 (S/A) (Demo. Weld)	6	0.15	1.28	0.010	0.012	0.20	0.66	0.06	0.48	0.05	0.02 V, <0.01 Sn
A543-1 (S/A)	6	0.10	0.84	0.006	0.008	0.35	0.83	2.40	1.10	0.01	0.02 V
A543-1 (S/A) (NRL Code N40)	2-1/4	0.07	1.23	0.007	0.006	0.51	0.71	2.07	0.96	0.04	0.05 V
A543-1 (S/A) (NRL Code N43)	2-1/4	0.07	1.21	0.007	0.006	0.49	0.80	2.07	0.93	0.26	0.04 V
9Ni-4Co-.20C (GTA)	2	0.15	0.39	0.005	0.003	0.17	10.00	1.0	0.60	0.11	0.08 V, 3.8 Co
12Ni-5Cr-3Mo(TIG)	4	0.01	0.02	0.010	0.002	0.14	11.00	5.07	2.78	...	0.48 Ti, 0.18 Al

^aCourtesy Armco Steel Corporation.

TABLE NRL-4 - HEAT TREATMENT OF PLATE, FORGINGS, AND WELD DEPOSITS

Material Type	Thickness (in.)	Heat Treatment ^(a)
<u>Plate</u>		
A302-B (ASTM Reference)	6	Austenitized 1650°F (899°C)-2 h, WQ; tempered 1200°F (649°C)-6 h, FC to below 600°F (316°C)
A533-B (Demo. Melt)	6	Austenitized 1675°F (913°C)-6 h, WQ; re-austenitized 1575°F (857°C)-6 h, WQ; tempered 1250°F (677°C)-6 h, FC; stress relief annealed (SRA) 1125°F (607°C)-2 h, FC; Re-SRA 1150°F (621°C)-8 h, FC to 600°F (316°C) at 90°F/h (50 C/h) max.
A543-1	8	Austenitized 1650°F (899°C)-8 h heating + 2 h hold, WQ 17 minutes; re-austenitized 1500°F (816°C)-8 h heating + 2 h hold, WQ 17 minutes; tempered 1185°F (640°C)-8 h heating + 2 h hold, WQ cold
A543-1	6	Austenitized 1675°F (913°C)-7.5 h, WQ; re-austenitized 1575°F (857°C)-7.5 h, WQ; tempered 1160°F (627°C)-6 h, WQ; SRA 1050°F (566°C)-2 h, AC; Re-SRA 1135°F (613°C)-6 h, AC
9Ni-4Co-.20C	2	Normalized 1650°F (899°C)-2 h, WQ; austenitized 1550°F (843°C)-2 h, WQ; tempered 1025°F (552°C)-4 h, AC; SRA 975°F (524°C)-6 h, FC at 100 F/h (56 C/h) max.
<u>Forging</u>		
12-6 PH (H1025)	4	Solution treated: 1900°F ±25°F (1038°C ±14°C)-0.5 h, OQ; hardened 1025°F ±15°F (552°C ±8°C)-4 h AC
12-6 PH (H1100)	4	Solution treated; 1900°F ±25°F (1038°C ±14°C)-0.5 h, OQ; hardened 1100°F ±15°F (593°C ±8°C)-4 h AC
<u>Weld Deposit</u>		
A533-B-2 (S/A) (Demo. Weld)	6	SRA 1150°F ±25°F (621°C ±14°C)-8 h; FC to 600°F (316°C) at 90°F/h (50°C/hr) max.
A543-1 (S/A)	6	Post-weld SRA: see Re-SRA of 6-in. A543-1 plate above
A543-1 (S/A) (NRL Code N40)	2-1/4	Post-weld SRA: 1150°F (621°C)-8 h, FC
A543-1 (S/A) (NRL Code N43)	2-1/4	Post-weld SRA: 1150°F (621°C)-8 h, FC
9Ni-4Co-.20C (GTA)	2	Post-weld SRA: see SRA of 2-in. 9Ni-4Co-.20C plate above
12Ni-5Cr-3Mo (TIG)	4	Post-weld marage: 900°F (482°C)-6 h, FC

^aWQ-water quenched
 OQ-oil quenched
 FC-furnace cooled
 AC-air cooled

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FIGURE NRL-2. Increase in Elevated Temperature YS with 550°F (288°C) Irradiation for the A302-B (ASTM Reference) Plate and for Low Copper Content A533-B Plates and Weld Deposit from Commercial-Scale Demonstration Tests. Results for specimens thermally conditioned at 550°F (288°C) are also shown (half filled symbols).

FIGURE NRL-3. Increase in Elevated Temperature YS with 550°F (288°C) Irradiation for A543 Class 1 Plates and Submerged Arc Weld Deposits. Half filled symbols refer to 550°F (288°C) thermal control tests. A 600°F (316°C) irradiation determination for one A543 plate is also shown.

75 to 550°F (24 to 288°C). The relationship is unchanged by fluence except for the yield strength elevation. Third, the slope of the yield strength versus temperature trend is about the same for all A302-B, A533-B, and A543-1 materials, but is somewhat less than the general slope of the trend for the higher strength alloys. Fourth, all data patterns infer that accurate projections of yield strength behavior for temperatures in the 75 to 550°F (24 to 288°C) range should be possible from only limited post-irradiation data.

Material intercomparisons indicate a significant detrimental effect of a high copper content on yield strength behavior with irradiation for Type A302-B, A533-B, and A543-1 materials. For the case of a low copper content, plates and weld deposits are shown to have the same yield strength elevation for a particular fluence. The A543-1 steel appears to experience a greater yield strength elevation than either A302-B or A533-B steels. On the other hand, the yield strength elevations for the high strength materials with exception of the 12-6PH alloy (H1100 condition) appear equal to or less than that for the A302-B steel. Of special interest to pressure vessel applications, it is noted that the yield strengths of the A302-B and A533-B plates and of the A533-B weld deposit after 2 to 2.5×10^{20} n/cm² are less than 110 ksi.

FIGURE NRL-4. Increase in elevated Temperature YS with 550°F (288°C) Irradiation Exhibited by the High Strength Materials. Half filled symbols refer to 550°F (288°C) thermal control tests.

Charpy-V upper shelf determinations for the plates, forgings, weld deposits are summarized in Table NRL-5. For the pre-irradiation condition, the upper shelf was reached at a temperature from 80 to 250°F (27 to 121°C) for a majority of the materials. After irradiation, most upper shelf temperatures fell between 250 and 400°F (121 and 204°C). Two notable exceptions

TABLE 5
 CHARPY-V UPPER SHELF PERFORMANCE OF PLATES, FORGINGS, AND WELD DEPOSITS
 BEFORE AND AFTER 550°F (299°C) IRRADIATION

Material	Fluence(a) (x10 ²⁰ n/cm ² >1 MeV)		C _V Upper Shelf Energy (ft-lb)		Post-irrad Temp (°F)	C _V Upper Shelf (approx.) (°C)
	φ≤	φ≤	Unirrad	Irrad		
<u>Plate</u>						
A302-B	1.6	1.7	81	56	<340	<171
(ASTM Ref)	6.5	6.8	81	33	440	227
A533-B-1	2.4	2.5	140	99	~350	~177
(Demo. Melt)						
A543-1 (8-in.)	1.6	1.7	72	46	<200	< 93
A543-1 (6-in.)	2.8	3.0	96	23	≥550	≥288
9Ni-4Co-.20C	2.0	2.1	76 ^c	55	<400	<204
<u>Forging</u>						
12-6 PH (H1025)	2.6	2.7	102 ^d	104	≤380	≤193
12-6 PH (H1100)	2.6	2.7	131 ^e	110	<300	<149
<u>Weld Deposit</u>						
A533-B-2	2.4	2.5	127 ^f	82	≤320	≤160
(Demo. Weld)						
A543-1 (6-in.)	2.8	3.0	102	45	~380	~193
A543-1 (2-1/4 in.)	1.2	1.3	81	58	~280	~138
(NRL Code N40)						
A543-1 (2-1/4 in.)	1.0	1.1	68	43	<420	<216
(NRL Code N43)						
9Ni-4Co-.20C	1.9	2.0	≥80 ^g	46	≥500	≥260
12Ni-5Cr-3Mo	0.96	1.0	84 ^h	66	~500	~260

^afluence, $\phi^{CS} > 0.1$ MeV = $2.15 \times \phi^{CS} > 1$ MeV

^cupper shelf level at 500°F (260°C); upper shelf level to 300°F (149°C) was 62 ft-lb

^dupper shelf level between 200°F (93°C) and 400°F (204°C)

^eupper shelf level at 400°F (204°C); upper shelf level to 200°F (93°C) was 124 ft-lb

^fupper shelf level at 550°F (288°C); upper shelf level to 200°F (93°C) was 118 ft-lb

^gweld deposit exhibited 80 ft-lb at 250°F (121°C) and rising

^hweld deposit exhibited 71 ft-lb at 200°F (93°C) and gradual rise to 84 ft-lb at 500°F (260°C)

were the A543 plates. Here, a difference in heat treatment condition (non-temper embrittled versus temper embrittled) and a difference in radiation embrittlement sensitivity (low versus high copper content) combined to produce a very large difference in post-irradiation upper shelf temperature for these materials. Eight material conditions resulted in shelf values of 50 ft-lb or higher; six material conditions resulted in shelf values less than 50 ft-lb.

The Ratio Analysis Diagram (RAD), developed at NRL, offers a simple, graphical method for assessing post-irradiation fracture resistance in the upper shelf level condition. Figure NRL-5 shows the RAD with the results of this investigation entered. The RAD suggests that, among the materials investigated, only the A533-B plate and weld deposit would exhibit plastic fracture behavior (very high toughness, plastic overload required for failure) in 6-in. thick section after a fluence of 2.5×10^{20} . (Note that this evaluation assumes uniform material properties through-thickness after irradiation.) At a 2.0×10^{20} fluence, the A302-B plate is shown to have potential for plastic fracture behavior with respect to the orientation tested (longitudinal); however, the weak orientation (transverse) of this plate probably would exhibit elastic-plastic-behavior i.e., intermediate toughness for the same fluence. Elastic-plastic behavior, nonetheless, is quite acceptable for many if not most service applications. It is noted that both the A533-B and A302-B material profited by having post-irradiation yield strength levels less than 110 ksi.

FIGURE NRL-5. Trend in Charpy-V Shelf Energy Reduction vs YS Increase for the Neutron-Irradiated ASTM A302-B Reference Plate and A533-Plate and Weld Metals. Shaded enclosures for A533 steel represent data for multiple grades and strength classes of thick section material before and after irradiation at 550°F (288°C) [3]. High fluence, 550°F (288°C) irradiation determinations by the present study are also shown (open square symbol).

Surprisingly high thick section fracture resistance was shown by the experimental 12-6PH alloy. The data for the H1025 and H1100 conditions indicate either plastic or high elastic-plastic toughness for a fluence of 2.7×10^{20} . Accordingly, this alloy holds significant promise for structural applications requiring high strength and high fluence resistance.

Data acquired for two of the materials (A302-B and 8-in. A543-1 plates) help verify a projected trend of C_v upper shelf energy versus yield strength change with irradiation. Reported trends for $<300^\circ\text{F}$ (149°C) irradiation are reproduced in Figures NRL-6 and 7.⁽³⁾ Filled points denote $<300^\circ\text{F}$ (149°C) exposures. Numbers adjacent to individual points indicate fluence in 10^{19} n/cm² >1 MeV. The open circle point in each figure refers to a moderate fluence irradiation at 550°F (288°C). Because of the superposition of these data on the trend line in each case, it had been suggested that shelf energy/yield strength changes with 550°F (288°C) irradiation follow the same damage path as that described for $<300^\circ\text{F}$ (149°C) irradiation. Entry of the results of the present study (open squares) in Figures NRL-6 and 7 gives direct support to the initial assumption.

Finally, the high upper shelf toughness retention of the A533-B plate and weld deposit offers a clear example of the benefit of minimizing impurity copper, phosphorus, and sulfur contents in reactor vessel steels as proposed by new ASTM and AWS specifications. The evidence against copper content in the low to medium strength steels is undisputable. (The mechanisms of copper-enhanced radiation embrittlement in lower strength bainitic alloys have also been studied and have been described.⁷⁾ However, it is noted that the 12-6PH alloy which had high upper shelf toughness after irradiation utilizes 3% Cu for precipitation hardening. Moreover, the data for this alloy indicate a difference in irradiation effect depending on heat treatment (and copper precipitation). It is proposed here that the present knowledge of the copper content contribution to radiation sensitivity might be expanded and refined by studies using the martensitic, 12-6PH materials.

C. TOUGHNESS OF IRRADIATED 316 SS FORGING AND WELD METAL USING THE J-INTEGRAL

F. J. Loss and R. A. Gray, Jr.

1. Background

The fracture toughness of austenitic stainless steel and nickel-base alloys for advanced fast breeder reactors is being characterized in terms of the J-Integral. These alloys, when employed at elevated temperatures, generally exhibit elastic plastic or fully plastic behavior. However, a high preservice toughness can be degraded by neutron irradiation during service. The J-Integral concept provides a means with which to define the initiation of crack extension in the elastic plastic regime and to relate this phenomenon to structural parameters used in design (e.g., critical flaw size and stress level). This procedure is based on a mathematical model for which experimental verification has not yet been fully achieved. Nevertheless, the toughness characterization of structural alloys must proceed simultaneously with experimental investigations designed to evolve standard measurement techniques for J and to define the effects of specimen geometry variations. The subject research explores the toughness of 316 SS forging and weld metal at elevated temperature. The J-Integral values are defined using a multi-specimen R-curve technique that is being evaluated as a measurement method.

FIGURE NRL-6. Trend in Charpy-V (C_V) Shelf Energy Reduction vs YS Increase of the 8-in. A543 Class 1 Plate with Neutron Irradiation [3]. A high fluence, 550°F (288°C) irradiation determination by the present study is also shown (open square symbol).

FIGURE NRL-7. The Ratio Analysis Diagram (RAD) illustrating the Pre- and Post-Irradiation Upper Shelf-Toughness Performance of the Several Materials. Data points for the low copper content A533-B plate and weld deposit (commercial-scale demonstration tests) indicate retention of plastic fracture behavior in thick section after $2.5 \times 10^{20} \text{ n/cm}^2 > 1 \text{ MeV}$ at 550°F (288°C).

2. Materials and Specimen Types

Weld metal test specimens were machined from a 1-in. thick weldment formed by joining 316 SS plates by submerged arc welding, using a Type 316 filler. (See Reference 8 for detailed description of welding procedures.) The weldment was specially prepared to simulate that used in the Army's MH-1A reactor pressure vessel. Specimens of forged 316 SS were prepared from a 3-1/8-in. thick ring forging nozzle cutout from the MH-1A vessel. Chemical and tensile properties are given in Table NRL-6.

Notched three-point bend specimens of dimensions: width (W) = 0.394 in., thickness (B) = 0.197 in., and length (L) = 2.165 in., were machined from both the weldment and forging. The weld metal specimens were oriented with the plane of the crack perpendicular to the surface and with crack propagation in the welding direction. A principal working direction for the forging was not obtained; the longitudinal specimen orientation therefore was arbitrarily chosen parallel to the surface and the crack propagation was parallel to the surface of the forging. The specimens were fatigue precracked at a ΔK maximum of $25 \text{ ksi}\sqrt{\text{in.}}$ using a zero-tension cycle in accordance with ASTM Standard E-399 procedures. Different weld metal specimen types were investigated having notch depth (a) to width ratios, a/W, from 0.2 to 0.5. The forging specimens had a nominal a/W of 0.35.

Weld metal specimens were irradiated in the Union Carbide Research Reactor at 500°F (260°C) to a fluence of $5.3 \times 10^{19} \text{ n/cm}^2 > 1 \text{ MeV}$. The forging specimens were irradiated in the Advanced Test Reactor at 530°F (277°C) to a fluence of $6.7 \times 10^{20} \text{ n/cm}^2 > 1 \text{ MeV}$. During irradiation the forging specimens were exposed to water, whereas the weld metal specimens were encapsulated in a helium environment. The testing was conducted statically using a three-point bend jig having a span of 1.758 in. between supports. The specimen load and midspan deflection were monitored on an X-Y recorder. All tests were conducted at the MH-1A operating temperature, i.e., 500°F (260°C). It is expected that J-Integral characterizations at this temperature can be used to define the material behavior at the low end of the temperature and fluence range to be experienced by LMFBR structural alloys.

3. Results

Specimens of a given a/W ratio were loaded to various fractions of the maximum load as determined from one of the specimens. This procedure resulted in the specimens undergoing different amounts of slow stable crack extension (Δa) with rising load which attaining the desired load or deflection. In preliminary interpretation of the data, no attempt was made to distinguish a stretch zone at the tip of the fatigue crack from the total crack extension values that are reported. The specimens were unloaded and heat tinted at 1100°F (593°C) and then broken open at room temperature. In this way, it was possible to measure the amount of stable growth that had taken place up to the point where the test was terminated. With the weld metal specimens, the slow crack extension could be distinguished from the fatigue pre-crack due to a difference in texture between these two regions. However, this difference in texture was extremely difficult to detect in the forged specimens. Consequently, it was necessary to ink stain the latter specimens before testing in order to mark the fatigue pre-crack region. The remaining heat tinted region was then attributed to slow crack extension that occurred with rising load during the course of the test.

TABLE NRL-6

CHEMISTRY AND MECHANICAL PROPERTIES OF 316 SS FORGING AND WELD METAL

Chemistry

Material	Composition (wt %)									
	C	Mn	P	S	Si	Ni	Cr	Mo	Co	Ta
316 SS Forging	0.06	1.51	0.014	0.010	0.57	10.80	17.60	3.00	0.01	0.09
316 SS S/A Weld	0.06	2.1	0.023	0.009	0.65	11.7	18.8	2.7	--	--
Mechanical Properties										
Material		Temp	Unirradiated		Irradiated ²					
		(°F)	YS(1)	TS(1)	YS(1)	YS(1)				
316 SS Forging		RT	39.7	80.5	77.7 ³	87.7 ³				
		500	28.7	66.7						
316 SS S/A Weld		500	44.4	68.3	73.4 ³	81.1 ³				

¹ ksi = 6.7×10^{20}

² Φ_{forging} = 5.3×10^{19} n/cm² >1 MeV

³ Φ_{weld}
data from rectangular compression plugs

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The J-Integral value was computed from the total area (A) under the load versus midspan deflection record (i.e., energy) using the approximate equation for a deeply notched beam in pure bending derived simultaneously by Rice⁽⁹⁾ and Srawley.

$$J = \frac{2A}{B(W-a)} \quad \text{Eq. 1}$$

In all cases, the deflections of the bend jig were negligible so that the area under the load versus deflection trace represents energy imparted to the specimen.

The use of Eq. 1 according to Rice requires a subtraction of the energy that would have been absorbed by an unnotched beam subjected to the same load as the test specimen; according to Srawley, this subtraction is not required. Investigators in this field have not yet determined which approach is the better. For a deeply notched beam ($a/W \geq 0.5$), however, the difference between the two approaches can be small. Using specimens of $a/W = 0.3$, the present investigation has defined a difference of approximately 7% between the two methods for unirradiated weld metal. However, this difference increases to as much as 50% for the irradiated weld specimens of $a/W = 0.3$ (20% for $a/W = 0.4$). These differences are more pronounced for low J values where the unnotched beam deflection is more significant than it would be with a specimen that tolerates substantial deflection in the absence of slow crack extension. Since this topic is not yet resolved, the results shown here do not include the unnotched beam correction.

The J-Integral toughness trends from irradiated and unirradiated weld metal specimens are represented in Figure NRL-8 as a function of the average slow crack extension at the specimen quarter points (i.e., an R-curve). Figure NRL-9 presents a similar comparison for the forging. In a strict sense, crack extension is not permitted by the J concept, since the J_{IC} value is defined as the toughness at crack initiation. This value is obtained here by extrapolating the R-curve to some near-zero crack extension such as may be acceptable in a valid* K_{IC} test (i.e., 2% crack extension). A 1% crack extension was arbitrarily chosen for this investigation. The J values as a function of Δa have been plotted using the original crack length (a) to the tip of the fatigue crack, rather than using the final length ($a + \Delta a$) when the test was terminated. The former method is used because lower (conservative) J values result. However, the difference between the two methods of plotting becomes very small at the point where J_{IC} is determined because Δa , by definition, is small.

4. Discussion

Figure NRL-8 shows that the J_{IC} for the irradiated weld is reduced to less than half of the unirradiated value when subjected to a fluence of $5/3 \times 10^{19} \text{ n/cm}^2 > 1 \text{ MeV}$. This degradation in toughness is potentially serious for a structure such as the MH-1A vessel. The weld in this vessel is expected to accumulate a lifetime fluence of the above magnitude. If one assumes that $J_{IC} = G_{IC}$ for plane strain conditions, then K_{IC} may be calculated from the following expression:

$$G_{IC} = \frac{(1-\nu^2)}{E} K_{IC}^2 \quad \text{Eq. 2}$$

where ν and E are Poisson's ratio and Young's modulus respectively. For a J_{IC} of 260 in.lb/in.

*Requirements for valid K_{IC} testing are given in ASTM Standard E-399.

FIGURE NRL-8. Trend of J-Integral Values with Slow Crack Extension for Weld Metal. The critical J, or J_{IC} , is defined by the intersection of the curve through the data (R-curve) and an arbitrarily chosen value of 1% crack extension. The arrows labeled Max. Load denote those points based on J computed using the specimen energy to maximum load. No trend in the data is evident as a function of a/W.

FIGURE NRL-9. Trend of J-Integral Values with Slow Crack Extension for Forging Material. The highest value on each curve was computed from the specimen energy to maximum load. For the unirradiated data, the two (square) points having a different W and a/W appear to be within the scatterband of the specimens having a/W = 0.35 and W = 0.394 in. A comparison with Fig. 8 illustrates the higher toughness of the forging in comparison to the weld metal.

for the irradiated weld metal, the equivalent K_{IC} is $86 \text{ ksi}\sqrt{\text{in}}$. According to ASTM E-399 thickness requirements, a plane strain K_{IC} value could be obtained with a 3.4-in. section for a material having the above toughness and a yield strength of 73.4 ksi as indicated in Table NRL-6. The thickness of the MH-1A vessel is 3-1/8-in. which, for all practical purposes, would meet the above thickness requirement. It must be remembered, however, that the weld in the vessel will not be uniformly embrittled to the value quoted above due to the self-shielding effect of the wall.

The J_{IC} of the forging material, illustrated in Figure NRL-9, is at least a factor of four higher than that of the weld in both the unirradiated and irradiated conditions.* Irradiation, however, has reduced the J_{IC} of the forging to one-third of its pre-irradiation value (3500 to 1000 in.lb/in.^2), but this still exceeds the pre-irradiation toughness of the weld metal (600 in.lb/in.^2). Even with the reduced toughness of the irradiated forging, one would expect this material to exhibit a high level of elastic plastic behavior when used in a structure.

The data in Figures NRL-8 and 9 illustrate a significant variation in J with crack extension. For example, the value of J associated with the crack extension near maximum load for the unirradiated forging (i.e., Δa equal to 0.035 in.) in Figure NRL-9 is more than twice as large as that associated with the first few mils of crack extension. Energy values associated with maximum load therefore appear to be unacceptable to establish J_{IC} when slow crack extension occurs. In practice, some crack extension must be permitted in order to define a critical value of J_{IC} at crack initiation, and 1% extension was arbitrarily chosen in this investigation. Additional research is necessary to evolve an acceptable means of defining "initiation."

For J_{IC} data to be considered valid, it is reasonable to require that certain minimum dimensions be maintained, as in the case of plane strain testing (i.e., ASTM Standard E-399). Landes and Begley,⁽¹¹⁾ on the basis of data from ferritic steels, have tentatively suggested that a , B , and W - a be greater than $25 J/\sigma_{flow}$ where $\sigma_{flow} = 1/2 (\sigma_{ys} + \sigma_{ts})$. For the specimens having $a/W = 0.5$, these conditions are essentially met for the weld metal but not for the forging. Under the preceding requirement, a valid J_{IC} for the forging material would necessitate specimen thicknesses of 0.3 and 1.83 in. for the irradiated and unirradiated condition respectively. Tests are in progress using 0.4-in. thick specimens to evaluate the thickness requirement for the forging in the irradiated condition. However, sufficient material does not exist with which to verify the stated thickness requirement for the unirradiated forging.

A final comment is in order concerning the geometry differences of the specimens tested. Current recommendations for J-Integral testing by others in the field suggest that a/W be maintained at 0.5 or greater in order to use the approximate expression for J (Eq. 1). The dimensions of the weld metal specimens were chosen to investigate the requirement. Within the limited data available, there appears to be no trend in the data of Figure NRL-8 with a/W . However, further research is required on which to base a firm conclusion.

*Note that the fluence of the forging is an order of magnitude above that of the weld.

D. MICROSTRUCTURE AND SWELLING OF EBR-II CONTROL ROD THIMBLE MATERIAL

D. J. Michel and H. H. Smith

1. Background

Liquid metal fast breeder reactors will utilize the austenitic stainless steels as structural components and fuel cladding. This application has necessitated the development of considerable information concerning the effects of elevated temperature fast neutron irradiation on the microstructure and properties of these steels. An important source of material for some of these investigations has been the structural components removed from EBR-II after attaining very high fast neutron fluences. Consequently, the swelling behavior, microstructure, and tensile properties of these components have been extensively evaluated.⁽¹²⁻¹⁹⁾ Included in these studies has been the recently reported NRL evaluation of the fatigue properties of annealed Type 304 stainless steel from EBR-II control rod guide thimble 5D3.⁽²⁰⁻²¹⁾

This evaluation has now been extended to include an examination of the microstructure and swelling of the fatigue tested thimble material by transmission electron microscopy (TEM) and immersion density methods.

2. Progress

The annealed 304 SS used was taken from flat number four of the upper section of EBR-II control rod guide thimble 5D3. This material was removed from the reactor after accumulating 42,321 megawatt-days of reactor exposure and a maximum calculated fluence of 1.6×10^{23} n/cm² >0.1 MeV. The axial irradiation temperature and fluence distributions for the material are shown in Figure NRL-10a and 10b, respectively.

The TEM and immersion density specimens were obtained from the unstrained end sections of previously prepared and tested fatigue specimens.⁽²⁰⁻²¹⁾ Prior to the TEM examination, the 3.2-mm square by 1-mm thick TEM specimens were reduced in thickness to 0.25-mm by careful, low-speed grinding. A twin-jet electropolishing technique⁽²²⁾ was found to produce excellent thin foils for subsequent TEM examinations conducted at 200 kV.

The specimens used for the immersion density measurements were approximately 3.2-mm wide by 25-mm long by 1-mm thick with a mass of approximately 0.5 gram. The specimens received no special surface preparation, except that all rough edges remaining from the cutting procedures were removed by lightly abrading the specimen edges. The method used to determine the density changes was similar to that described by Ratcliffe.⁽²³⁾

The TEM and immersion density results obtained from the present work are summarized in Table NRL-7. From the table, it is evident that the effects of both irradiation temperature and neutron fluence strongly influence the mean void diameters, number densities, and the resultant swelling. This is also evident in Figure NRL-11, where examples of the void structures observed by TEM are illustrated.

The mean void diameter and void number density, determined from the TEM data, are shown in Figures NRL-12a and 12b as functions of irradiation temperature. The void volumes calculated from the respective void diameters and number densities are shown in Figure NRL-12c. For comparison, the swelling determined by the immersion density method is shown in Figure NRL-10c as a function of specimen location in EBR-II. It is seen from Table NRL-7 and Figures-10 and

12 that the maximum swelling occurred concurrently with the maximum observed void diameter, slightly above the maximum fluence location, and slightly below the maximum observed void number density. The peak seen in Figure NRL-12a also indicates the substantial effect of neutron fluence on void diameter. In all cases, the present void parameters are consistent with previously published results for neutron-irradiated 304 SS.⁽¹²⁻¹⁹⁾

FIGURE NRL-10. Irradiation Temperature (a), Neutron Fluence (b), and Swelling (Immersion Density Change) (c) Profiles for EBR-II Control Rod Thimble 5D3, Flat Number Four.

The dislocation densities estimated from the TEM data are also given in Table NRL-7. It is seen that these data indicate little variation with either irradiation temperature or neutron fluence. This suggests that recovery of the dislocation structure has occurred as the result of thermal exposure during the fatigue tests, especially for material represented by TEM specimens taken from regions of maximum neutron fluence.

No attempt was made to obtain complete quantitative data for the dislocation loops observed throughout the TEM specimens. However, qualitative observations indicate that the number density of small dislocation loops was considerably higher for those specimens irradiated at temperatures between 370 and 380°C than for those specimens irradiated at temperatures between 450 and 480°C. Within the higher irradiation temperature range, the Frank faulted loop concentrations were found to vary from $2 \times 10^{15}/cm^3$ to $3 \times 10^{14}/cm^3$ with decreasing neutron fluence. In this same temperature range, evidence of loop growth resulting from thermal recovery also was observed for those specimens exposed to the 593°C fatigue test temperature.

TABLE NRL-7
SUMMARY OF MICROSTRUCTURE DATA FOR SOLUTION-TREATED 304 SS
EBR-II CONTROL ROD THIMBLE 5D3

Specimen #	Distance Above or Below Core Midplane ^(a) (in.)	Neutron Fluence ^(b) (total n/cm ²)	Irradiation Temp ^(b) (°C)	Test Temp ^(c) (°C)	Dislocation Density (#/cm ²)	Mean Void Diameter (Å)	No. of Voids Measured	Void Number Density (#/cm ³)	Void Volume (Calculated) ΔV/V (%)	Swelling ^d (Immersion Density Change) -Δρ/ρ (%)
1	-12.25	1.3x10 ²²	371	427	-	102	519	1.1x10 ¹⁶	0.83	1.56
2	- 9.60	6.0x10 ²²	371	593	4.0x10 ¹⁰	131	238	1.3x10 ¹⁶	1.75	2.03
3	- 6.50	1.0x10 ²³	371	427	-	-	-	-	-	2.84
4	- 3.80	1.3x10 ²³	382	593	1.3x10 ¹⁰	162	413	8.2x10 ¹⁵	2.23	3.99
5	- 1.60	1.5x10 ²³	388	427	1.9x10 ¹⁰	180	1153	8.0x10 ¹⁵	3.06	7.54
7	7.20	1.0x10 ²³	438	-	1.1x10 ¹⁰	228	590	2.5x10 ¹⁵	1.94	2.69
8	9.90	7.0x10 ²²	449	593	1.1x10 ¹⁰	188	392	4.1x10 ¹⁵	1.95	1.73
9	13.00	1.4x10 ²²	460	427	9.0x10 ²	180	502	7.4x10 ¹⁴	0.35	1.08
10	15.70	9.0x10 ²¹	471	593	1.5x10 ¹⁰	179	676	2.8x10 ¹⁴	0.21	0.97
15	-12.25	1.3x10 ²²	371	593	9.6x10 ⁹	134	250	1.6x10 ¹⁵	0.25	1.29
16	- 9.60	6.0x10 ²²	371	427	4.1x10 ¹⁰	116	636	5.8x10 ¹⁵	0.68	2.11
22	5.00	1.3x10 ²³	427	427	3.4x10 ¹⁰	260	243	8.7x10 ¹⁵	9.96	10.25
23	7.20	1.0x10 ²³	438	593	1.7x10 ¹⁰	232	432	5.3x10 ¹⁵	4.37	3.79
24	9.90	7.0x10 ²⁰	449	427	1.9x10 ¹⁰	201	566	3.6x10 ¹⁵	2.10	2.18
25	13.00	1.4x10 ²²	460	593	1.7x10 ¹⁰	210	715	9.2x10 ¹⁴	0.65	1.63
26	15.70	9.0x10 ²¹	471	427	1.0x10 ¹⁰	186	658	4.4x10 ¹⁴	0.23	0.74
34	3.00	1.8x10 ²³	415	-	-	264	602	8.8x10 ¹⁵	10.40	10.40
35	-13.50	9.0x10 ²³	371	-	-	98	453	1.2x10 ¹⁶	0.93	1.19

^aLocation of fatigue specimens. Specimens for immersion density and TEM displaced slightly in location.

^bCalculated from EBR-II supplied data.

^cTemperature at which fatigue tests were conducted.

^dDensity of unirradiated thimble stock determined to be 7.902 gm/cm³ at 22°C.

$T_{\text{irrad}} = 427^{\circ}\text{C}$, $\phi t = 1.3 \times 10^{23} \text{ n/cm}^2$

$T_{\text{irrad}} = 385^{\circ}\text{C}$, $\phi t = 1.5 \times 10^{23} \text{ n/cm}^2$

$T_{\text{irrad}} = 460^{\circ}\text{C}$, $\phi t = 1.35 \times 10^{23} \text{ n/cm}^2$

$T_{\text{irrad}} = 471^{\circ}\text{C}$, $\phi t = 9.0 \times 10^{21} \text{ n/cm}^2$

FIGURE NRL-11. TEM of Typical Voids Observed in 304 SS from EBR-II Control Rod Thimble 5D3, Flat Number Four.

FIGURE NRL-12. Mean Void Diameter (a), Void Number Density (b), and Swelling (c), as a Function of Irradiation Temperature for 304 SS from EBR-II Control Rod Thimble 5D3, Flat Number Four.

Precipitates, identified as $M_{23}C_6$, were observed at the grain boundaries of all specimens. Both large and small $M_{23}C_6$ precipitates also were observed through the matrix of the material. The large matrix precipitates were frequently surrounded by a high concentration of small voids which formed a gradient away from the precipitates. It is believed that this void size and concentration gradient directly reflects the effects of solute segregation in the vicinity of the large precipitates. The small $M_{23}C_6$ precipitates within the matrix were observed to be randomly distributed and generally not associated with the voids.

The immersion density results presented in Figure NRL-10c are compared in Figure NRL-13 with the density decrease calculated according to the empirical expression developed for use with the LIFE II fuel element performance code.⁽¹⁶⁻²⁴⁾ The comparison shows that the calculated density decrease is greater than the experimental values, except for those specimens irradiated at the low temperatures below the core midplane. At the position of maximum swelling, the calculated density decrease is seen to overestimate the observed decrease by nearly 50%. This suggests that the empirical expression will require revision to more accurately represent the swelling observed in the presently available 304 SS thimble material.

Further characterization of the material from thimble 5D3 is presently in progress. When completed, all results will be presented in a future NRL report.

FIGURE NRL-13. Measured and Calculated Density Decrease as a Function of Specimen Core Position for 304 SS from EBR-II Control Rod Thimble 5D3, Flat Number Four.

II. RADIATION DAMAGE MECHANISMS

A. THE DOSE DEPENDENCE OF NICKEL ION DAMAGE IN NICKEL

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1. Background

The irradiation of metals with high-energy charged particles is now being extensively used as a means of rapidly simulating the effects of long-term neutron irradiation at elevated temperatures. The simulation is qualitatively accurate in most cases in that the same types of defect clusters are formed by both types of irradiation. However, our present understanding of the effects of such factors as (10^3 to 10^6)-fold increases in damage rate, the much smaller volume of material that is damaged in a charged-particle irradiation, and differences in the displacement cascades produced by different bombarding particles do not allow us to confidently predict the quantitative relationships between charged-particle and neutron-irradiation results.

The aim of the present research is to provide a systematic evaluation of the effects of irradiation parameters on the microstructures produced by heavy-ion irradiation. High-purity nickel, bombarded with self-ions, was selected for this investigation to avoid some of the complications, such as phase instability, that can occur in commercial alloys. These results have been compared with the predictions of theoretical models for void nucleation and growth and with previously-published data on neutron- and charged-particle-irradiated nickel. These comparisons have provided insights into the prediction of neutron-irradiation results by heavy-ion irradiation and have suggested additional experiments to further increase our understanding of charged-particle simulation of neutron damage. The effects of irradiation temperature and dose rate were discussed in a previous progress report.⁽²⁵⁾ The following section extends these analyses to the effect of damage density (expressed as displacements per atom or dpa) at one temperature.

2. Experimental Results

The procedures used for preparation, bombardment, and analysis of the specimens were the same as described previously.⁽²⁵⁾ Damage levels of 4, 13, 40, and 130 dpa were studied for specimens bombarded at a temperature of 575°C and a damage rate of 7×10^{-2} dpa/sec. The void and dislocation structures were examined following bombardment by TEM. The void and dislocation structures observed in these specimens are shown in Figures NRL-14 and 15 respectively. The two types of defects are shown in separate micrographs because different diffraction conditions were required for effectively imaging them. The void density, mean void diameter, swelling, and dislocation density determined for these specimens are given in Table 8. It can be seen that the void density decreased and their diameter increased with increasing displacement density. The dislocation density was fairly constant up to 40 dpa, but had decreased significantly by 130 dpa. However, the nature of the dislocation structure observed at 4 dpa was somewhat different from that seen at the higher displacement densities. The structure at 4 dpa consisted largely of loops and relatively isolated segments while at 13 dpa and higher, there was much more of an interconnected network.

TABLE NRL-8

EFFECT OF IRRADIATION DOSE ON VOIDS AND
DISLOCATIONS IN NICKEL-ION-IRRADIATED NICKEL

Displacement Density (dpa)	Void Density (cm ⁻³)	Mean Void Diameter (Å)	Dislocation Density (cm ⁻²)	Swelling (%)
4	1.1×10^{16}	75	2.7×10^{10}	0.28
13	6.9×10^{15}	130	2.0×10^{10}	0.90
40	5.5×10^{15}	150	2.4×10^{10}	1.23
130	6.6×10^{14}	380	7.8×10^9	2.16

3. Discussion

To analyze the material processes controlling the void formation in these ion irradiation experiments, the data have been compared with theoretical models for the nucleation and growth of voids. The analyses of the high- and low-dose-rate temperature-dependence experiments, which appear in detail elsewhere,⁽²⁵⁾ indicated the following: the swelling was principally limited by void growth below the temperature of maximum swelling and by void nucleation above

FIGURE NRL-14. The Dependence of the Void Structure on Irradiation Dose in Nickel Irradiated at a Damage Rate of 7×10^{-2} dpa/sec and an Irradiation Temperature of 575°C. The damage levels in the specimens were: (a) 4 dpa, (b) 13 dpa, (c) 40 dpa, and (d) 130 dpa.

FIGURE NRL-15. The Dependence of the Dislocation Structure on Irradiation Dose in Nickel Irradiated at a Damage Rate of 7×10^{-2} dpa/sec and an Irradiation Temperature of 575°C. The damage levels in the specimens were: (a) 4 dpa, (b) 13 dpa, (c) 40 dpa, and (d) 130 dpa.

that temperature: the shift of the low temperature limit for void formation between neutron irradiation⁽²⁶⁾ and high-dose-rate ion irradiation could be predicted by void growth theory,⁽²⁷⁾ but the shifts in the peak swelling temperature and the high temperature limit could not be predicted by this theory; the shift of the low temperature limit between ion irradiations differing by a factor of 100 could also be predicted by void growth theory. In the following paragraphs, these analyses are extended to the displacement density dependence, and implications of the results for the prediction of swelling in neutron-irradiated metals are discussed.

Since the data in Table NRL-8 indicated that the void number density decreased with increasing displacement density above 4 dpa, implying that void nucleation had essentially stopped by this damage level, the analysis was limited to a consideration of void growth only. For the case of a metal containing no precipitates, the Brailsford and Bullough treatment of void growth theory⁽²⁷⁾ calculates the swelling rate as

$$\frac{d}{dt} \left(\frac{\Delta V}{V} \right) = \left\{ D_V C_V - D_i C_i - D_V \bar{C}_V^{(\text{void})} \right\} 4\pi \rho_V r_V \quad \text{Eq. 3}$$

where $\Delta V/V$ is the fractional volume change due to the voids, D_V and D_i are the diffusion coefficients of vacancies and interstitials, C_V and C_i are the vacancy and interstitial concentrations, ρ_V is the void number density, r_V is the mean void radius, and $\bar{C}_V^{(\text{void})}$ is the thermal equilibrium vacancy concentration at the surface of a void. The point defect concentrations are calculated by the defect balances:

$$K + K_{th} - D_V C_V k_V^2 - \alpha C_V C_i = 0 \quad \text{Eq. 4a}$$

$$K - D_i C_i k_i^2 - \alpha C_V C_i = 0 \quad \text{Eq. 4b}$$

where K is the atomic displacement rate, K_{th} is the thermal vacancy production rate, and α is the recombination coefficient.

The sink strengths for vacancies and interstitials, k_V^2 and k_i^2 are given by:

$$k_V^2 = Z_V \rho_d + 4\pi \rho_V r_V \quad \text{Eq. 5a}$$

$$k_i^2 = Z_i \rho_d + 4\pi \rho_V r_V \quad \text{Eq. 5b}$$

where $\rho_d = \rho_d^N + \rho_d^L$ is the total dislocation density, assumed to be made up of network dislocations (ρ_d^N), which emit thermal vacancies, and dislocation loops (ρ_d^L) which do not, and Z_V and Z_i are dislocation sink strength constants for vacancies and interstitials. The thermal vacancy production rate is then calculated by

$$K_{th} = D_V \left[Z_V \rho_d^N C_V^e + 4\pi \rho_V r_V \bar{C}_V^{(\text{void})} \right] \quad \text{Eq. 6}$$

where C_V^e is the thermal equilibrium vacancy concentration in a specimen without loops or voids. In practice, the effect of having $\bar{C}_V^{(\text{void})} > C_V^e$, expressing the fact that voids would shrink under thermal equilibrium, is the same as the effect of having $\rho_d^L \neq 0$. Therefore, for ease of discussion, we will henceforth set $\bar{C}_V^{(\text{void})} = C_V^e$ and let ρ_d^L be some fraction of the total dislocation density.

The vacancy and interstitial diffusion coefficients are calculated by

$$D_V = D_V^0 \exp (-E_V^m/k_B T) \quad \text{Eq. 7a}$$

$$D_i = D_i^0 \exp (-E_i^m/k_B T) \quad \text{Eq. 7b}$$

where D_V^0 and D_i^0 are the diffusion pre-exponentials, E_V^m and E_i^m are the activation energies for vacancy and interstitial motion, T is the absolute temperature, and k_B is Boltzmann's constant. In the calculations discussed below, the materials parameters for nickel were taken as: $E_V^f = E_V^m = 1.4$ eV, $E_i^m = 1.1$ eV, $D_V^0 = D_i^0 = 0.1$ cm²/sec, $\alpha/D_i = 10^{16}$ cm⁻², $\rho_d^L = 0.01 \rho_d$, and $Z_V = 1$. The dislocation bias for interstitial capture, $Z_i - Z_V$, will be specified with the individual results.

The vacancy sink strengths represented by the voids and dislocations as a function of displacement density at 575°C are shown in Figure NRL-16. The sink strengths were both essentially constant up to 40 dpa, and then both decreased approximately a factor of 3 by 130 dpa. The ratio $4\pi\rho_V r_V/\rho_d$ was 2.5 ± 0.6 for all damage levels investigated. If the materials parameters remained constant for all damage levels, the void growth theory would predict that the swelling rate would be constant from 4 to 40 dpa and then would decrease by 130 dpa. The experimental swelling data, however, are not entirely consistent with this prediction, as shown in Figure NRL-17. The slope of line (a) represents the swelling rate calculated using the 4 dpa void and dislocation parameters and $Z_i - Z_V = 0.06$. The slope of line (b) represents the swelling rate calculated using the 40 dpa void and dislocation parameters and $Z_i - Z_V = 0.01$. The slope of line (c) represents the swelling rate calculated with the 130 dpa void and dislocation parameters and $Z_i - Z_V = 0.01$. While there are not sufficient data points to define the linear swelling region, the present results are consistent with the form of the displacement density dependence observed by Ryan and Taylor⁽²⁸⁾ for swelling in nickel-ion-irradiated nickel, which was a rapid initial swelling followed by a linear dependence of swelling on damage level. Furthermore, the data above 13 dpa are consistent with the 0.01 to 0.02 dislocation bias for interstitial capture that has been previously proposed in the literature.⁽²⁷⁻²⁹⁾

The fact that it was necessary to set $Z_i - Z_V = 0.06$ at 4 dpa to get approximate agreement with the void growth theory implies that some factor affecting void growth rate must have been changing between 4 and 13 dpa. Two possible explanations of this behavior are that either the steady-state vacancy and interstitial concentrations had not yet been established, so the steady-state theory was not valid, or that the effective dislocation bias was higher for the 4 dpa microstructure than for the structures at 13 dpa and higher. Wiedersich⁽²⁹⁾ has suggested that an estimate of τ_{SV} , and the relaxation time for the point defect concentrations to come to steady state during irradiation, is

$$\tau_{SV} = (3\overline{x^2}/b^2)/\nu_V \quad \text{Eq. 8}$$

where $\overline{x^2}$ is the mean square sink separation, b is the vacancy jump distance, and ν_V is the vacancy jump frequency. At 575°C, for the void and dislocation parameters observed at 4 dpa, this gives $\tau_{SV} \approx 8 \times 10^{-2}$ sec; which is the equivalent of approximately 6×10^{-3} dpa at the dose rate employed in this experiment. Thus, it does not appear plausible that this transient effect could explain the rapid initial swelling.

FIGURE NRL-16. The Vacancy Sink Strengths Calculated for the Void and Dislocation Structures Observed in Nickel as a Function of Irradiation Dose at a Damage Rate of 7×10^{-2} dpa/sec and an Irradiation Temperature of 575°C .

FIGURE NRL-17. Comparison of the Dose Dependence of Swelling Observed in Nickel and Swelling Rates Calculated by the Void Growth Theory.[27] The data points are the swelling values measured for specimens irradiated at a damage rate of 7×10^{-2} dpa/sec and an irradiation temperature of 575°C.

Considering a change in the dislocation bias during irradiation, Figure NRL-14 showed that the dislocation structure at 4 dpa consisted largely of loops and isolated segments, while the structure at the higher displacement levels consisted primarily of an interconnected network. It is possible that the bias of the 4 dpa structure could be different from that of the structure observed for 13 dpa and above. This conclusion would be in qualitative agreement with the model developed by Wolfer⁽³⁰⁾ for the point defect biases of sinks in irradiated metals, but a more detailed analysis will be required to fully understand these results.

The shape of the ion-dose dependence of swelling (initial rapid swelling followed by approximately linear swelling at a considerably slower rate), raises some interesting questions regarding the relationships among swelling results from different bombarding particles. This form of the ion damage curve, which has been previously reported for both nickel-ion⁽²⁸⁾ and carbon ion⁽³¹⁾ irradiations of nickel, contrasts with the results of Norris⁽³²⁾ for 1 MeV electron irradiations of nickel, which showed swelling to have a power law dependence on displacement density:

$$\frac{\Delta V}{V} \propto (D)^n \quad \text{Eq. 9}$$

where D is the displacement density in dpa, and the value of n was 2.2 ± 0.2 below 10 dpa and was approximately 1 from 10 to 40 dpa. Above 40 dpa, the swelling saturated at approximately 8%. The limited amount of fluence dependence data that is available for neutron-irradiated nickel suggests a power law dependence, as in Eq. 9, with n decreasing from a value of about 3 at 4×10^{17} n/cm² to a value of 1 at 1×10^{20} n/cm².⁽³³⁾ In another study, the linear dependence of swelling on fluence was found to extend to 4×10^{21} n/cm².⁽³⁴⁾ These results, however, were all at very low displacement densities (total dpa) in relation to the ion and electron irradiation experiments, so more thorough analyses of data for all the bombarding particles would be necessary to determine how the various results relate to one another.

4. Conclusion

Analysis of the void and dislocation structures as a function of irradiation dose in nickel-ion-irradiated nickel indicated that the swelling rate decreased significantly between 4 and 13 dpa. This decrease could not be explained by a change in the vacancy sink strengths of the voids on dislocations. A possible explanation of the variation in swelling rate is a change in the dislocation structure leading to a decrease in the bias of the dislocations for interstitial capture.

B. STUDIES OF THE MOBILITY OF HELIUM IN 316 SS AND ALUMINUM

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The current studies were undertaken to gain some understanding of the mobility of helium in metals and indirectly its influence on helium embrittlement and void nucleation. Thin foils of 316 SS and aluminum were implanted with helium in the NRL Cyclotron using a beam of 70 MeV alpha particles and a series of aluminum foils of appropriate thickness to degrade the beam so as to deposit the helium in the foils. Specimen temperature during implantation was kept below 200°C by bonding the foils to a water cooled copper block with silver conducting paint. The foils were subsequently annealed at progressively higher temperatures and examined by TEM to determine if helium bubbles had formed and to characterize the bubble populations. An α - α

elastic scattering technique previously described⁽³⁵⁾ was also used to study the motion of helium in aluminum during annealing.

1. Experimental Observations - 316 SS

A total of 25 specimens of 316 SS has been implanted with helium in the concentration range 20 to 70 ppm and annealed for 1 hour at temperatures of 600, 700, 800, 900, 1000, and 1100°C to examine the microstructure developed during annealing. No damage was visible in the as-irradiated condition, but small defects showing black-white contrast typical of small dislocation loops were seen after the 600°C anneal. These grew to readily resolvable 500Å loops with a reduction in density during the 700°C anneal. All the dislocation loops had annealed out at 800°C and a few ~25Å diameter bubbles were observed. These grew in size and decreased in density at 900, 1000, and 1100°C. Figure NRL-18 is a micrograph of the bubbles after 1000° anneal. Quantitative TEM measurements of the results are summarized in Table NRL-9. Each data point includes several observations of different areas of the specimen weighted by number of defects observed. The presence of dislocations was found to strongly perturb the size and concentration of bubbles in a given volume, although the total helium concentration calculated for that volume was not influenced.

The helium concentration of the bubbles was calculated using the modified Van der Waals equation of state,

$$m_i = \frac{2/3 \pi \gamma d_i^2}{kT + (4 b\gamma/d_i)} \quad \text{Eq. 10}$$

m_i , the helium content for the median size bubble, was determined for each size class measured on the particle size analyzer. These values were then multiplied by the frequency factor for the size class and summed over all size classes. Values of γ determined for 304 SS⁽³⁶⁾ used in the calculations were 2630 ergs/cm² at 800°C, 2440 at 900°C, 2250 at 1000°C, and 2050 at 1100°C. Values of the Van der Waals constant, b , taken from the tables of Ref. 37 were $b = 9.123, 8.882, 8.658, \text{ and } 8.450 \text{ ml/mole}$ at 800, 900, 1000, and 1100°C respectively. Some uncertainty exists in the exact helium concentration implanted because of problems with beam drift during the implantation. Specimens implanted to a nominal 65 ppm level were analyzed by Atomics International and found to contain from 55 ± 2 to 68 ± 2 ppm helium. In spite of uncertainties in the implanted helium concentration in these specimens, the TEM results showed good reproducibility from sample to sample and the calculated concentrations using the Van der Waals equation of state are considered to be within $\pm 20\%$ of the actual concentration.

2. Experimental Observations - Aluminum

The mobility of helium has been studied in both cold-worked and annealed aluminum foils. Sample A1-1 was implanted with 1000 ppm helium and annealed in place in the scattering chamber using resistance heating of the copper frame in which the sample was mounted. The temperature was monitored by a thermocouple spot welded to the frame. Helium concentration profiles were determined for the as-implanted condition and after 1-hour anneals at 400, 500, and 600°C, as shown in Figure NRL-19. No change was noted between the as-implanted condition and the 400°C anneal, but the peak concentration decreased by 26% after the 500°C anneal and by 66% after the 600°C anneal. TEM observations of areas adjacent to the foil center showed quite large bubbles (mean diameter 545Å) in a matrix still containing many dislocations.

FIGURE NRL-18. Typical Area Showing Helium Bubbles in 316 SS After a 1-hour Anneal at 1000°C. Mean diameter was 36Å, bubble density was $1.7 \times 10^{14} \text{ cm}^{-3}$, and helium concentration was 29 ppm. Bubbles were imaged under weakly diffracting contrast conditions and 1.5 to 2.3 μm overfocus.

TABLE NRL-9
OBSERVATIONS OF BUBBLES

Specimen Number	History	Mean Diameter (Å)	Density (cm ⁻³)	Helium Content (ppm)	
				Calc.	Implanted
SS-4	1 h - 800 C	<u>316 SS</u> ~25	$\sim 4.4 \times 10^{14}$	2.2	20-60
SS-5	1 h - 900 C	39.1	1.75×10^{15}	35.3	20-60
SS-6	1 h - 900 C	63.6	2.47×10^{14}	18.6	20-60
SS-7	1 h - 1000 C	112.4	1.08×10^{14}	28.1	20-60
SS-8	1 h - 1000 C	85.8	1.66×10^{14}	28.9	8-32
SS-9	1 h - 1100 C	151.5	5.38×10^{13}	25.2	8-32
		<u>Aluminum</u>			
A1-1	Cold Work, 1 h 400, 500, 600 C	545.6	2.41×10^{13}	233	{ 370-740 125-250 ^b
A1-2	Cold Work, 1 h 582 C	57.1 ^a	1.72×10^{16}	1280	{ 2840 ~1100-1400 ^b
A1-3	1 h - 382 C	17.3	8.4×10^{16}	208	95
A1-4	1 h - 477 C	30.9	3.0×10^{15}	71.1	95
A1-5	1 h - 582 C	107.7 ^c 164.6 ^a 182.0	1.54×10^{14} 4.70×10^{13} 5.80×10^{13}	66.1 40.3 61.8	{ 56 110

^aTruncated octahedron, $r_{\text{eff.}} = 1.113 r_{\text{meas.}}$ ^bConc. after anneal. ^cCuboidal $r_{\text{eff.}} = 1.241 r_{\text{meas.}}$

Specimen A1-2 was also a cold-worked foil, but was taken from a stack of degrader foils used as a beam stop in one experiment. TEM observations of this foil, implanted with 2840 ppm helium and annealed at 582°C for 1 hour, showed a few large bubbles on grain boundaries but, as shown in Figure NRL-20, consisted primarily of dislocation free areas with a high density of octahedral bubbles with a mean diameter of 57Å. The calculated helium content was 1280 ppm. This foil was not water cooled during implantation and it is likely that a temperature rise occurred which could have resulted in recovery of the cold-worked structure and void formation during implantation.

Specimens A1-3, A1-4, and A1-5 were annealed aluminum implanted with 95 to 110 ppm helium. The helium concentration profile was measured before and after annealing and, contrary to the behavior in the cold-worked foils, no motion of helium was seen for 1-hour anneals at 382, 477, and 582°C. TEM results on A1-3 showed bubbles of 15 to 30Å diameter present after an anneal of 382°C with $\bar{d} = 17.3\text{Å}$. These bubbles were only visible in thin sections <1000Å, with a through focus series $\sim \pm 7700\text{Å}$. Helium concentration calculated from the bubble population was greater than that implanted, so it is suspected these are actually voids formed during implantation. Specimen A1-4 annealed at 477°C had a high density of bubbles with a mean diameter

FIGURE NRL-19. Helium Bubbles Observed in Aluminum, Specimen A1-2, Implanted with 2840 ppm Helium, and Annealed at 582°C for 1 hour. Mean diameter of the circle projected on the octahedra was 57.1 Å, bubble density was $1.7 \times 10^{18} \text{ cm}^{-3}$, and helium concentration was 1280 ppm. Bubbles were imaged in the weakly diffracting contrast condition and 1.5 to 2.3 μm overfocus.

of 31Å. Specimen Al-5 annealed at 582°C showed much larger bubbles, obviously influenced by the presence of dislocations introduced in the foil during handling. Three areas analyzed show approximately the same helium concentration, but appreciable scatter in the bubble sizes and densities. Helium concentrations in all specimens except Al-1 were calculated from the Van der Waals equation of state using a surface energy of 1000 ergs/cm². Westmacott, et al⁽³⁸⁾ found a value of 1140 ± 200 ergs/cm² for the surface energy of aluminum from shrinkage of voids in the temperature range 150 to 200°C.

FIGURE NRL-20. A Plot of Surface Diffusivity, D_s , Values Calculated from Gruber's Equation Relating Bubble Size for Various Annealing Times, Temperatures, and Helium Concentrations with Surface Diffusivity vs $1/T$. Linear relations indicate a fit to the model and the slopes yield surface migration energies.

3. Discussion

These experiments on annealing of helium-implanted foils have helped clarify several aspects of the mobility of helium in metals. Some conclusions can be drawn directly from the observations while others, such as the mechanism of bubble growth, require a more detailed analysis. Among the more significant observations was the finding that helium loss from annealed foils was negligible during anneals up to $0.84 T_m$. Calculations of the helium content of observable bubbles using the Van der Waals equation of state showed that, within the limits of experimental error, most of the helium precipitated in bubbles at about $0.65 T_m$. The presence of dislocations and grain boundaries strongly influenced the growth of bubbles. At low temperatures, the dislocations may be serving as a source of vacancies so the bubbles can reach equilibrium size, but this cannot be true at the higher temperatures, because the total helium content in regions containing dislocations and large size bubbles was found to be the same as

adjacent regions which had few dislocations. Several possibilities exist including one-dimensional motion of bubbles along dislocations, high diffusivity paths along the dislocation, and collection of helium during climb of dislocations.

The loss of helium from the CW aluminum foils during annealing was in striking contrast to its immobility in a well annealed foil. The sweeping bubbles during recovery and recrystallization thus appears to be a much more important transport mechanism than diffusion in CW material. Finally, irradiation temperature influences bubble nucleation and growth. The abundant supply of vacancies during irradiation would permit reaching equilibrium size at a lower temperature and increase diffusivity for a substitutional mechanism. However, it would also involve nucleation of voids on bubble nuclei⁽³⁹⁾ since, for example, specimen Al-3 had a calculated helium concentration greater than that implanted. Additional experiments are needed to investigate this possibility.

These experiments also provide the necessary data to compare the bubble growth models for collision and coalescence of bubbles during random walk migration and by Ostwald ripening. The random walk migration of bubbles over distance $\bar{R}$ can be described in terms of a bubble diffusion coefficient D_B , $\bar{R}^2 = 6 D_B t$. Motion of the bubble can be controlled by either vapor transport of material across the bubble, transport by diffusion through the lattice or motion of atoms on the surface of the bubble. Surface diffusivity is generally considered to be the controlling process in the range investigated in these experiments. Gruber⁽⁴⁰⁾ has shown the surface diffusivity to be related to the bubble diffusivity by the equation,

$$D_s = \frac{1}{.301} \left(\frac{r}{a_o} \right)^4 D_B.$$

He has further characterized the bubble size distribution which evolves from collision and coalescence of the bubbles at equilibrium with an internal pressure of an ideal gas. The mean radius of this distribution is related to the surface diffusivity by the equation,

$$D_s = \frac{\gamma}{nkT a_o^4 t} \left[\frac{\bar{r}}{1.30} \right]^5,$$

where n is the helium content in atoms/cm³, a_o is the lattice parameters, $\bar{r}$ is the mean radius of the bubble population, and the other parameters have their usual meaning. D_s values for the various bubble populations characterized in these experiments have been calculated and plotted as Arrhenius equations in Figure NRL-20 to examine the consistency of the data. The results will be discussed further below.

The other process of bubble growth, Ostwald ripening, is a consequence of the difference in solubility of helium in equilibrium with large bubbles and small bubbles so that large bubbles grow at the expense of small ones. The rate controlling process is diffusion of helium through the lattice. Markworth⁽⁴¹⁾ has analyzed the evolution of the bubble size distribution for this process and derived equations relating to the volume diffusivity to the mean radius of the bubble population,

$$D_V K = \frac{4(\bar{r})^2}{3kT t},$$

where K is the Sieverts' law coefficient, $C_r = KP_r$, relating the concentration of gas in equilibrium with a bubble with internal pressure, P , for radius, r . K was included as part of the argument because it was unknown, but, as a constant, would still permit testing the applicability of the model. Results from these experiments are plotted in Figure NRL-21.

FIGURE NRL-21. A Plot of $D_v K$ vs $1/T$ to Test the Ostwald Ripening Model of Bubble Growth as Related to the Size of Bubbles by Markworth (see text). A linear relation is expected for agreement with the model and the slope of the lines yields a value for the migration energy of helium through the lattice.

The data for vanadium discussed in the previous report⁽³⁵⁾ are found to give an excellent fit for the surface diffusivity model and yield a migration energy of 2.4 eV for the surface migration energy. A similar plot for the Ostwald ripening model did not fit as well. The maximum slope gave a migration energy of 1.0 eV. The data for 316 SS shows considerable scatter for the points at 900 and 1000°C. This is believed to be a consequence of the influence of dislocations on the growth rate of bubbles. When the high values are discarded for these temperatures, a good fit to a straight line is obtained with a surface migration energy of 4.2 eV. The fit to the Ostwald ripening model had a reasonable fit to the 1100, 1000, and 900°C points with a 1.8 eV migration energy. The aluminum results from these experiments as well as the range of values found in the experiments of Chen and Cost for various annealing times at 550°C are plotted. The aluminum data have considerable scatter, with a sizable discrepancy between the results from Al-2 and Al-5 at 582°C. It is suspected that the results of Al-2 were affected by void nucleation during the implant, while those from Al-5 were too high because of dislocations in the sample. The point from Al-3 at 382°C is also suspected of being too high because of void nucleation. The line arbitrarily drawn through the points would give a surface migration energy of 2.1 eV. There was no obvious pattern to the aluminum data when plotted on the Ostwald ripening model.

The results of these experiments support the surface diffusivity model of bubble growth because the data fit the model more closely and because realistic values are obtained for surface migration energies. It has been suggested that the surface migration energy should be approximately the binding energy of an atom to the surface.⁽⁴³⁾ Thus, for face-centered-cubic metals, a value two-thirds of the heat of sublimation has been found for copper and aluminum. The present data for aluminum are $\sim 2/3 \Delta H_s$, while vanadium, a body-centered-cubic metal, is $\sim 1/2 \Delta H_s$. The stainless steel data seem high by this criteria, being just under the sublimation energy of nickel. By contrast, the migration energies calculated from the slopes of the $D_V K$ versus $1/T$ plots for Ostwald ripening are too high for migration of helium as an interstitial and too low for either the mutual diffusion process described by Wilson and Bisson⁽⁴⁴⁾ or diffusion as a substitutional atom.

Our current assessment of helium mobility in metals is that in experiments in which helium is implanted at low temperatures and then annealed at higher temperatures, diffusion of atomic helium occurs over short distances to nucleate bubbles, and these bubbles grow by collision during random migration. Imposition of a stress or thermal gradient will add a bias to the direction of motion. Sweeping of bubbles by dislocations is an important transport process for carrying bubbles to grain boundaries and out of the samples. Alloys strengthened by precipitates should retard this transport process.

Under irradiation, helium is probably formed in the interstitial position and will move rapidly as an interstitial until a vacancy, void, or other helium trap is encountered or, in the case of low energy alpha bombardments, it is lost to the surface. Once trapped at a vacancy, the helium atom combines with the vacancy and becomes a substitutional diffusion or the mutual diffusion mechanism.⁽⁴⁵⁾ Helium may be displaced into the interstitial position again by a displacement cascade or by encountering a self-interstitial which recombined with the vacancy and forces the substitutional helium back into the lattice.

4. Conclusions

This study of the mobility of helium using α - α elastic scattering and TEM in 316 SS and aluminum has shown that: a) helium precipitates in bubbles which grow to detectable sizes at about $0.65 T_m$ and these bubbles grow by collision and coalescence during random walk migration. The bubble migration appears to be controlled by diffusion of atoms on the surface of the bubble; b) there is very little loss of helium from the foils up to temperatures of $0.85 T_m$ or above, i.e., the mean free path for atomic migration of helium to bubbles was less than to the surface; c) the most significant losses of helium were found in CW samples in which helium bubbles were apparently swept out of the sample during annealing of the dislocation structure.

III. ARMY REACTOR STUDIES

A. EVALUATION OF TENSILE SPECIMENS FROM SURVEILLANCE CAPSULES OF MH-1A REACTOR

J. R. Hawthorne and C. Z. Serpan, Jr.

The MH-1A reactor vessel surveillance program provides specimen capsules at accelerated exposure, vessel wall, and above-core (thermal control) locations. Details of the program have been documented elsewhere.⁽⁴⁶⁾ Results from specimen capsules removed during the Spring 1969 and Spring 1971 refueling outages have also been documented. A third series of five

capsules removed during the Spring 1973 refueling outage was received recently for post-irradiation evaluation. The capsules represented all three surveillance locations and had been installed in the reactor prior to its initial startup. Capsule disassembly and the evaluation of Charpy-V specimens from each capsule were reported previously,⁽⁴⁷⁾ tensile specimen test results are now available and are listed in Table NRL-10.

TABLE NRL-10
TENSILE PROPERTIES OF MH-1A VESSEL FORGING
(SURVEILLANCE CAPSULE IRRADIATIONS)

Capsule Number	Surveillance Location	Test Temp. (°F)	Yield Strength (ksi)	Tensile Strength (ksi)	Specimen Number
A2-1	Accelerated	75 ^a	98.3	113.3	30
		500 ^b	69.7	86.1	29
A1-1	Accelerated	75	84.2	103.7	9
		500	56.6	78.6	8
		500	57.5	78.5	11
W2-2	Vessel Wall	75	56.0	89.6	27
		500	34.3	68.7	25
		500	35.4	68.3	26
W2-1	Vessel Wall	75	49.2	85.7	6
		500	33.8	70.1	5
CT-3	Above-Core (Thermal Control)	500	31.2	65.7	12
		500	29.9	64.7	13

^a24°C
^b260°C

Observed yield and tensile strength trends are illustrated in Figures NRL-22 and 23. Closed symbols refer to 75°F (24°C) determinations; open symbols refer to 500°F (260°C) tests. Neutron fluences received at individual irradiation locations, in general, decrease from left to right. Actual neutron fluences are not yet available. Several observations are possible from the data.

- 1) Data for the accelerated irradiation locations show an appreciable increase in both yield and tensile strength. On the other hand, data for the vessel wall exposure did not indicate a significant irradiation effect. Specimens from the above-core (thermal control) locations also did not show an effect of exposure on strength.
- 2) Data trends indicated by 75°F (24°C) versus 500°F (260°C) tests are comparable for the three surveillance locations.
- 3) The highest fluence condition (accelerated exposure, capsule A2-1) did not elevate the yield strength above 100 ksi.

The strength determinations for the MH-1A vessel forging, together with previously reported notch ductility determinations, should preclude any concern for fracture resistance of the vessel forging at this point in service.

FIGURE NRL-22. Increase in Yield and Tensile Strength of MH-1A Reactor Vessel Forging Produced by Surveillance Capsule Irradiations (Core I, II, and III exposure).

FIGURE NRL-23. Yield and Tensile Strength of MH-1A Reactor Vessel Forging after Irradiation in Vessel Surveillance Capsules (Core I, II, and III exposure).

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I. LMFBR PROGRAM--EFFECTS OF COMPOSITION ON THE SWELLING AND POST-IRRADIATION MECHANICAL PROPERTIES OF STAINLESS STEEL

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The purpose of this work is to elucidate the role of composition on the swelling behavior and irradiation-induced mechanical-property changes in austenitic stainless steels. During this reporting period, we have been extending our observations through an examination of samples that had been irradiated in the EBR-II subassembly X100A. Analysis of these results to assess the influence of composition on alloy behavior has continued.

A. EXPERIMENT DESCRIPTION

1. Titanium Additions

Alloys having the base composition of 316 SS with titanium additions of 0.23, 0.33, 0.46, and 0.60 wt % (designated R1, S1, T1, and U1, respectively) were produced by vacuum melting. Titanium additions were made to a master alloy. Approximately 50-lb (22-kg) ingots were cast between each titanium addition. The ingots were reduced by the vendor to 0.5-in. (12.7-mm) diameter rods. Chemical analysis of the rods is given in Table ORNL-1. Also included in Table 1 are the compositions of three other alloys for which swelling data are available and comparisons can be made. The successive melting resulted in a continual decrease in manganese. The variation of other elements except titanium is probably within experimental error of the analysis. The 0.5-in. (12.7-mm) diameter rod was annealed 1 hr at 1200°C, swaged to 0.395 in. (10.3-mm) in diameter (at room temperature), annealed 1 hr at 1200°C and swaged 0.280 in. (7.11mm) in diameter. Test specimens with 0.125-in. (3.18-mm) diameter by (1-in.) (25.4-mm) long sections were machined from a portion of the rod at this point. These specimens were then annealed 1 hr at 1050°C.

An additional alloy containing 0.26 wt. % Ti (designated heat Z0) was produced by vacuum melting. The ingot was extruded to 2.0-in. (50.8-mm) diameter bar cold swaged to 1.2 in. (30.5-mm) in diameter, annealed 1 hr at 1200°C, swaged to 0.83 in. (21.1-mm) in diameter and annealed 1 hr at 1050°C. Specimen holders of the design shown previously⁽¹⁾ were then machined. A portion of the annealed bar was swaged to 0.74 in. (18.8-mm) in diameter (~20% reduction in area) and specimen holders were machined from this cold-worked bar. The specimen holders were used for post-irradiation density and TEM examination.

2. Molybdenum Variations

Alloys having the nominal composition of 316 SS except for variations in molybdenum content were produced by vacuum melting. The 0.5-in. (12.7-mm) casting was swaged at room temperature 0.25-in. (6.4-mm) diameter rod with an intermediate anneal at 1200°C. The final swage

*Operated by Union Carbide Corporation for the U. S. Atomic Energy Commission

reduced the diameter from 0.35-in. (9.0-mm) to 0.250-in. (6.4-mm). The chemical analysis of the as-fabricated alloys are listed in Table ORNL-2. Samples were machined from each of the three alloys, annealed 1 hr at 1050°C, and irradiated in common specimen holders in ERB-II sub-assemblies X100 and X100A. Samples were irradiated to fluences of 2.1 and 5.6×10^{26} n/cm² (>0.1 MeV) at calculated temperatures of 500 and 600°C.

TABLE ORNL-1
CHEMICAL ANALYSIS OF EXPERIMENTAL ALLOYS FOR TITANIUM VARIATION STUDY

Alloy Designation	Composition, wt%										
	C	Cr	Mn	Mo	N	Ni	Si	Ti	B	S	P
R1	0.06	17	0.5	2.5	0.0055	12	0.4	0.23	0.0007	0.012	0.010
S1	0.05	17	0.4	2.5	0.0060	12	0.4	0.33	0.0007	0.010	0.009
T1	0.05	17	0.3	2.4	0.0046	13	0.4	0.46	0.0005	0.009	0.009
U1	0.04	17	0.2	2.5	0.0048	13	0.4	0.60	0.0005	0.004	0.008
ZO	0.05	17	1.1	2.4	0.012	13	0.4	0.26			
MS (Hi Purity) ^a	0.005	17.5		2.8	0.0003	14.4	0.01		<0.00001	0.0020	0.006
DO Commercial ^a	0.050	17.8	1.92	2.6	0.0560	13.0	0.75	0.05		0.016	0.013
Brager ^b	0.052	17.8	1.9	2.33	0.041	13.55	0.30		<0.0005	0.02	0.12

^aJ. M. Leitnaker, E. E. Bloom, and J. O. Stiegler, "The Effect of Minor Constituents on Swelling in Stainless Steel," *J. Nucl. Mater.* 49: 57-59 (1973).

^bH. R. Brager and J. L. Straalsund, "Defect Development in Neutron Irradiated Type 316 Stainless Steel," *J. Nucl. Mater.* 46: 134-58 (1973).

TABLE ORNL-2. CHEMICAL ANALYSIS OF EXPERIMENTAL ALLOYS
FOR MOLYBDENUM VARIATION STUDY

Designation	Composition, wt %								
	Mo	Ni	Cr	Mn	C	Si	S	N	Fe
A2	0.21	11.9	16.7	1.99	0.07	0.84	0.017	0.002	Bal
O2	2.46	11.2	16.9	2.02	0.07	0.92	0.016	0.002	Bal
P2	5.15	11.0	17.4	1.96	0.07	0.81	0.018	0.002	Bal

3. Irradiation Conditions

All specimens were irradiated in row 2 of the EBR-II in subassembly X10-A. Irradiation temperatures were in the range of 450 to 700°C. Temperatures above the ambient sodium coolant temperature were obtained by means of a gas gap between the surface of cylindrical specimen holders and the inside of the containment tube. In some cases, changes in temperature occurred due to differential swelling between the containment tube and specimen holder. The temperatures were calculated using both the initial (pre-irradiation) and final (post-irradiation) dimensions of the containment tubes and specimen holders. In those cases where the calculations showed that a change in temperature greater than 10°C had occurred during irradiation, the irradiation temperatures are given as "Initial Temperature"/"Final Temperature" (for example, 675/715°C).

The accuracy of the calculated temperatures is dependent on the temperature and is estimated to be $\pm 15^{\circ}\text{C}$ at 450°C , $\pm 30^{\circ}\text{C}$, and $\pm 50^{\circ}\text{C}$ at 750°C .

4. Post-irradiation Examination

The post-irradiation examination is continuing. Immersion density measurements are being made and the microstructure of the material in the as-irradiated condition is being characterized by TEM examination of samples removed from the shoulders of the mechanical property specimens prior to testing and from the specimen holders. Quantitative measures of defect concentrations are obtained by determining foil thicknesses by stereomicroscopy.

Post-irradiation creep-rupture tests are performed in lever-arm creep machines. Specimen elongation as a function of time is determined by a linear differential transformer that measures the relative movement of the upper and lower specimen grips. Creep-rupture tests are also performed on unirradiation specimens in the as-heat-treated and as-heat-treated-and-aged conditions.

B. RESULTS

1. Effects of Titanium

A portion of these results have been reported previously,⁽²⁾ but will be included here for completeness.

The variation in irradiation-produced microstructure with titanium content is discussed in detail elsewhere.⁽²⁾ The observations will be summarized in the present report. Data on void concentrations, size distributions and calculated volume changes are given in Table ORNL-3. For purposes of comparison, it is important to note that the samples from heats MS, S1, T1, and U1 were irradiated in the same sample holder and thus were at the same temperature and fluence. The other samples were irradiated in separate holders at other axial and radial positions of the reactor and thus there may be differences in temperature and fluence compared to the above mentioned⁽⁴⁾ alloys due to experimental uncertainties. Alloys with titanium contents in the range 0.23 to 0.60 wt % exhibited increasing swelling with increasing titanium. In the samples irradiated at 450°C to $1.0 \times 10^{26} \text{ n/cm}^2$, the trend was present, but was probably within the experimental error. At 500 and 600°C and $2.0 \times 10^{26} \text{ n/cm}^2$, the trends were outside of the experimental error and such that plots of swelling vs temperature actually cross, as shown in Figure ORNL-1a. Swelling at low or zero titanium can be inferred from results of Brager and Straalsund⁽⁴⁾ or from a standard heat of 316 SS (heat D0) irradiated in the same sub-assembly⁽⁵⁾ used for the presently described irradiations. In either case, at this fluence level and these temperatures, a minimum in the swelling vs titanium curve exists. At 500°C heat D0 exhibited lower swelling than predicted from the results of Brager and Straalsund.⁽⁴⁾ This difference may arise from differences in silicon content. The average void diameter was nearly independent of titanium content, and the concentration of voids increased with increasing titanium. In the various alloys, the dislocation structures were similar for each irradiation temperature, with the exception of the high-purity alloy. At 500°C , faulted dislocation loops and a network were present except for the high-purity alloy, that contained only a network. At 600°C , only a network structure was present in all alloys. In samples containing faulted loops, it was found, in agreement with the results of Brager⁽⁶⁾ on standard 316 SS, that the different $\{111\}$ planes had different concentrations of loops. Also of interest

TABLE ORNL-3

TABULATION OF RESULTS OF TEM EXAMINATION

Sample ^a	Irradiation Conditions		Voids Counted	Volume Examined (m ³)	Void Concentration			Volume Averaged Diameter			Average Diameter			Void Fraction		
	Temperature (°C)	Fluence (n/m ²)			(voids/m ³)	Std. Dev.	Std. Dev. of Mean	(nm)	Std. Dev.	Std. Dev. of Mean	(nm)	Std. Dev.	Std. Dev. of Mean	(%)	Std. Dev.	Std. Dev. of Mean
RI-88 ^b	450	1.9×10^{26}			68×10^{19}						17.2			0.20		
MS-18 ^c	450	1.0			71			38.9						2.18		
SI-38	450	1.0	631	4.35×10^{-18}	14.5	2.88	1.44	22.3	0.7	0.4	21.8	0.7	0.3	0.084	0.020	0.009
TI-39	450	1.0	1469	10.9	13.0	4.02	1.64	24.2	1.0	0.4	23.7	1.0	0.4	0.094	0.023	0.009
UI-39	450	1.0	673	4.86	13.7	1.90	0.95	23.9	0.8	0.4	23.4	0.8	0.4	0.097	0.010	0.005
Brager ^d	450	1.0			21.5						21.0			0.119		
RI-127 ^b	515	2.3			1.8						31.3			0.035		
MS-1 ^c	500	2.0			39			54.0						3.23		
SI-30	500	2.0	266	7.22	3.88	1.2	0.45	27.5	2.1	0.8	26.7	2.2	0.8	0.042	0.013	0.005
TI-31	500	2.0	562	15.1	3.77	0.84	0.37	31.7	0.9	0.4	30.6	0.6	0.3	0.062	0.012	0.005
UI-31	500	2.0	412	5.03	8.22	0.88	0.62	29.8	0.8	0.6	28.9	0.7	0.5	0.114	0.012	0.008
Brager ^d	500	2.0			13.8						36.8			0.429		
DO ^{e,b}	500	2.0			3.7			35.3 ^f			34.3			0.10		
MS-2 ^c	600	2.0			6.4			114.0						5.03		
SI-31	600	2.0	176	59.3	0.288	0.112	0.042	52.4	6.3	2.4	50.0	6.1	2.3	0.024	0.016	0.006
TI-32	600	2.0	162	19.9	0.844	0.120	0.084	70.6	4.1	2.9	68.0	3.6	2.6	0.160	0.045	0.032
UI-32	600	2.0	581	11.2	5.28	0.42	0.21	56.3	2.4	1.2	53.3	2.2	1.1	0.493	0.054	0.027
Brager ^d	600	2.0			1.66						52.9			0.161		
DO ^{e,b}	600	2.0			0.74			72.4 ^f			70.1			0.18		

^aCompositions are given in Table 1.5.

^bSample was not irradiated in this experiment, but was obtained previously. It is included for comparison.

^cData taken from our previous work, "The Effect of Minor Constituents on Swelling in Stainless Steel," *J. Nucl. Mater.* 49: 57-66 (1973).

^dComputed from equations given by H. R. Brager and J. L. Straalsund, "Defect Development in Neutron Irradiated Type 316 Stainless Steel," *J. Nucl. Mater.* 46: 134-58 (1973).

^eThese samples are commercial grade type 316 stainless steel and the values have been adjusted from our previous work, footnote c, to stated temperature by using Brager and Straalsund's equations, footnote d. The observed value was divided by the B-S value at the observed condition and then multiplied by the B-S value at the desired condition. The comparison is thus easier but the interpolation procedure may introduce errors.

^fEstimated.

was the observation that, even though the swelling varied considerably between the high-purity alloy and the alloys containing various amounts of titanium, within experimental uncertainties, the dislocation line density was constant. Immersion density results for the same alloys at a fluence of $4.4 \times 10^{26} \text{ n/cm}^2$ are plotted in Figure ORNL-1 (b). A minimum in swelling is again present, possibly at a slightly higher titanium content than the lower fluence. Results on heat Z0 (0.26% Ti) irradiated in the annealed condition to 3.5 and $7.8 \times 10^{26} \text{ n/cm}^2$ are plotted in Figure ORNL-2. The temperature-dependence of swelling at the higher fluence differs between the standard and the titanium-containing alloy. In the latter, the swelling appears to be weakly dependent on temperature and the temperature for peak swelling is about 100°C higher.

FIGURE ORNL-1. Effect of Titanium Additions on Irradiation-Induced Swelling of 316 SS. (a) $2 \times 10^{26} \text{ n/m}^2$ and (b) $4.4 \times 10^{26} \text{ n/m}^2$.

Immersion density results for samples of 316 SS + 0.23 wt % Ti irradiated at 500 and 600°C in the 20% CW condition are shown in Table ORNL-4. At $3.0 \times 10^{26} \text{ n/cm}^2$, the 20% CW structure exhibited a small density decrease at 500°C and no change at 600°C . Microstructures after irradiation to $6.6 \times 10^{26} \text{ n/m}^2$ are shown in Figure ORNL-3. At 500°C , voids were present in clusters or patches, and these patches were estimated to occupy about 10% of the sample volume. At 600°C , no voids were observed. Recovery of the CW structure into a cell structure was apparent.

The effect of titanium content on the creep-rupture properties is illustrated by the data presented in Table ORNL-5. In the annealed condition, strength properties were highest for the alloys containing 0.23 and 0.33 wt % Ti. Ductility as measured by total creep elongation was quite high for all alloys. Aging for 200 hr at 650°C prior to test caused a marked decrease in the strength of all alloys including the standard type 316 SS. In the aged condition the alloys with 0.23, 0.33, and 0.45% Ti exhibited lower strength than the standard or 0.60 wt % Ti compositions. In general, aging tended to produce increased ductility. Additional results on the effects of thermal aging on the properties of standard 316 and the 316 + 0.23 Ti alloy, given in Table ORNL-6, indicate that after aging at 550°C the titanium-containing alloy retained

a strength advantage (at least for aging times of 4000 hr); while after aging at 650°C, the strength properties of the two alloys were approximately equal. The standard alloy was slightly stronger at a high stress (see Table ORNL-5), and the 316 + 0.23 Ti alloy was slightly stronger at a lower stress.

FIGURE ORNL-2. Swelling of 316 and TiM 316 SS Irradiated in the Annealed Condition.

TABLE ORNL-4

IMMERSION DENSITY CHANGE IN 316 + 0.26% Ti SS
 IRRADIATED IN THE 20% CW CONDITION

Irradiation Temperature (°C)	Fluence, $>0.1 \text{ MeV}$ (n/m^2)	Density Change (%)
500	3.0×10^{26}	+0.17
600	3.0	+0.10
500	6.6	-0.23
600	6.6	0.00

The creep-rupture properties of the various alloys after irradiation in the annealed condition at 500 and 600°C to $2 \times 10^{26} \text{ n/m}^2$ are listed in Table ORNL-7. The commercial 316 and

FIGURE ORNL-3. Microstructures of TiM 316 SS Irradiated at (a and b) 500°C and (c) 600°C to 6.6×10^{26} n/m². At 500°C, patches or clusters of voids were present. At 600°C, no voids were observed.

316 + 0.23 Ti alloys were irradiated in separate capsules at a calculated temperature of 580°C instead of 500°C as for the other samples. Unirradiated creep-rupture properties were not evaluated at 550°C and 45 ksi (310 MPa) because of the very long rupture times at this stress level. Using a parameter $[\theta = t_r \exp(-95,000/RT)]$ where t_r is the rupture life, R the gas constant and T the temperature (K) to correlate results of tests at other temperatures and stresses, we estimate that the unirradiated rupture life of the commercial 316 and 316 + 0.23 Ti would be 2500 and 35,000 hr, respectively. Irradiation, therefore, produces a substantial reduction of rupture life. Using a Dorn parameter $[\rho = \dot{\epsilon} \exp(95,000/RT)]$ where $\dot{\epsilon}$ is the minimum creep rate, the creep rates of the unirradiated alloys are estimated to be 8×10^{-4} and 8×10^{-5} for the commercial 316 and 316 + 0.23 Ti alloys. The decrease in rupture life as a result of irradiation is mainly associated with an increase in the minimum creep rate. Alloys containing 0.23 and 0.33 wt % Ti exhibited the highest total creep elongation. In these two alloys one-fourth to one-half of the time was spent in tertiary creep. This latter observation also applies to the higher titanium levels. The results given in Table ORNL-6 suggest that the rupture life increases with the titanium content, reaching a plateau between 0.33 and 0.46% Ti. The commercial 316 and 316 + 0.23% Ti samples were irradiated at 580°C, however, as compared to 500°C for the other alloys. The higher creep rates (i.e., an effect of thermal aging as discussed in previous paragraphs) and thus the observed larger reduction in rupture life. At 650°C and 25 ksi (172 MPa) strength and ductility occurred at 0.23 and 0.33 wt % Ti. For these samples, the irradiation temperatures were closer to a common temperature of 600°C. Using the results of Table ORNL-5, the rupture life was reduced by factors of 2 and 4.5 for the commercial 316 and 316 + 0.23% Ti alloys, respectively. For the 316 + 0.23% Ti alloy, this reduction was less than occurred as a result of aging 4000 hr at 650°C.

TABLE ORNL-5
VARIATION OF THE CREEP-RUPTURE PROPERTIES OF
TYPE 316 SS WITH TITANIUM CONTENT

Sample	Alloy	Rupture Life (hr)	Creep	
			Minimum Rate (%/hr)	Total Elongation (%)
Annealed 1 hr at 1050°C, tested at 650°C and 35 ksi (241 MPa)				
Standard 316		150	0.15	62
R1	316 + 0.23 Ti	582	0.003	47
S1	316 + 0.33 Ti	436	0.006	31
T1	316 + 0.46 Ti	125	0.034	42
U1	316 + 0.60 Ti	82	0.048	43
Annealed 1 hr at 1050°C, aged 2000 hr at 650°C, tested at 650°C and 35 ksi (241 MPa)				
Standard 316 ^a		35	0.8	56
R1	316 + 0.23 Ti	23	1.6	67
S1	316 + 0.33 Ti	17	1.7	60
T1	316 + 0.46 Ti	5	4.1	48
U1	316 + 0.60 Ti	32	1.0	75

^aStandard 316 was aged 4000 hr at 650°C instead of 2000 hr.

TABLE ORNL-6
EFFECT OF AGING 4000 HR AT 550 AND 650°C ON THE CREEP-RUPTURE
PROPERTIES OF 316 SS AND 316 + 0.23% Ti ALLOY

Temperature (°C)	Stress		As-Annealed			Aged		
			Rupture Life (hr)	Minimum Creep Rate (%/hr)	Creep Elongation (%)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Creep Elongation (%)
	(ksi)	(MPa)						
			Type 316 Stainless Steel					
550	60	413.7	164	0.03	26	264	0.02	17
650	25	172.4	668	0.034	57	551	0.037	47
			Type 316 + 0.23% Ti Alloy					
550	60	413.7	2181		30	1412	0.0007	30
650	25	172.4 ^a	~7000	~0.001	~50	754	0.036	56

^aExtrapolated from tests at stresses in the range 40-30 ksi (275.8-206.8 MPa).

TABLE ORNL-7
VARIATION OF CREEP-RUPTURE PROPERTIES OF 316 SS
WITH TITANIUM CONTENT AFTER IRRADIATION IN EBR-II

Alloy	Rupture Life (hr)	Minimum Creep-Rate (%/hr)	Elongation at Beginning of Tertiary Creep (%)	Total Creep Elongation (%)
Irradiated at 500°C to 2.0×10^{26} n/m ² (>0.1 MeV), tested at 550°C, 45 ksi (310.3 MPa)				
Commercial 316 ^a	452	2.6×10^{-3}	1.4	1.4
316 + 0.23 Ti	1930	3.6×10^{-3}	3.6	16.2
316 + 0.33 Ti	4236	7.4×10^{-4}	0.8	17.5
316 + 0.46 Ti	5567	3.6×10^{-4}		
316 + 0.60 Ti	5336	1.3×10^{-4}	0.3	4.0
Irradiated at 600°C to 2.0×10^{26} n/m ² (>0.1 MeV), tested at 650°C, 25 ksi (172.4 MPa)				
Commercial 316 ^b	347	3.3×10^{-2}	3.8	21.6
316 + 0.23 Ti ^b	1590	1.1×10^{-2}	6.0	27.8
316 + 0.33 Ti	1055	3.4×10^{-3}	1.1	18.2
316 + 0.46 Ti	533	4.4×10^{-3}	1.0	13.7
316 + 0.60 Ti	668	3.0×10^{-3}	0.6	16.5

^aIrradiated at 580°C to 2.7×10^{26} n/m².

^bIrradiated at 560-630°C to 2.3×10^{26} n/m².

Similar to the effects observed in 304 SS⁽⁷⁾ the addition of titanium to 316 SS produces a marked decrease in the tendency for grain-boundary crack formation in the unirradiated condition, as illustrated in Figure ORNL-4. In the standard alloy, cracks were generally observed along the entire gage length of the sample. As can be seen, the propensity for crack formation in the 316 + 0.23% Ti alloy was much reduced. The only cracks (or cavities) observed occurred near the fracture surface. The observed effect is not a result of grain refinement, as can be seen from examination of the sample given the 1100°C pre-test annealing treatment. Fractures of the 316 + 0.60% Ti alloy were similar to those found in the 316 + 0.23% Ti alloy.

FIGURE ORNL-4. Creep-Rupture Fracture in 316 and Ti-Modified SS. Standard 316 SS was tested at 650°C and 30 ksi (206.8 MPa). Ti-modified 316 was tested at 650°C and 35 ksi (241.3 MPa) after annealing 1 hr at 1050 and 650°C and 37.5 ksi (258.6 MPa) after annealing 1 hr at 1100°C.

After neutron irradiation, grain-boundary cracking and mixed inter-transgranular fractures were observed (Figure ORNL-5), but extensive propagation of individual cracks did not occur. The high ductility of the 0.23 and 0.33 wt % Ti alloys at 550°C as compared to the commercial alloy is mainly attributable to extensive tertiary creep. In the commercial alloy, little tertiary creep occurs; apparently a crack once formed propagates rapidly to cause failure. In the 316 + 0.23% Ti alloy cracks have formed (Figure ORNL-5) but have propagated slowly, allowing time for additional creep deformation.

FIGURE ORNL-5. Ti-Modified 316 SS (316 + 0.23% Ti) Irradiated at 580°C to 2.3×10^{26} n/m² (>0.1 MeV) and Creep Tested at 600°C and 40 ksi (275.8 MPa).

2. Effects of Molybdenum Concentration

Precipitate phases in unirradiated samples that had been annealed 1 hr at 1050°C and cooled in the water-cooled cold zone of the furnace were electrolytically extracted at 1.5 V in 10% HCl-90% CH₃OH. The precipitate was collected by centrifugation, weighed and x-rayed. The alloys containing 0.23 and 2.45 wt % Mo contained a low concentration of extracted precipitate (<0.1 wt %, except near the surface of the sample, where it was as high as 0.7 wt %). The sample containing 5.15 wt % Mo contained 10-13 wt % precipitate, however. X-ray examination revealed only weak and unidentified peaks from the precipitate extracted from the 0.21 and 2.46 wt % Mo alloys. The precipitate from the high-molybdenum alloy was similar to that of Cr-Fe Sigma phase. Spectrophotometric analysis of the supernate from the 0.21 and 2.46 wt % Mo alloys gave good agreement with the bulk analysis. We will thus be able to determine the major element composition of the matrix of the 5.5 wt % Mo alloy by difference when analytical data are available.

The density changes in samples irradiated to $2.1 \times 10^{26} \text{ n/m}^2$ were less than $\pm 0.1\%$. TEM of these samples is in progress. Density changes after irradiation to $5.6 \times 10^{26} \text{ n/m}^2$ are listed in Table ORNL-8. At 500°C, the swelling increased slightly (the differences being within the $\pm 0.1\%$ uncertainty of the density measurements) between 0.21 and 2.46 wt % Mo. The alloy with 5.5% Mo definitely exhibited higher swelling than the lower molybdenum contents. At 600°C, swelling increased monotonically with molybdenum, the effect being most pronounced in the range 0.21 to 2.46 wt %, where the density decrease increased from 0.66 to 4.34%.

TABLE ORNL-8
EFFECT OF MOLYBDENUM CONTENT ON RADIATION-
INDUCED DENSITY CHANGES IN 316 SS

Molybdenum Content (wt. %)	Irradiation Temperature (°C)	Fluence, >0.1 MeV (n/m^2)	Immersion Density Change (%)
0.21	500	5.6×10^{26}	-1.28
2.46	500	5.6	-1.37
5.15	500	5.6	-2.45
0.21	600	5.6	-0.66
2.46	600	5.6	-4.34
5.15	600	5.6	-6.43

C. SUMMARY AND DISCUSSION

1. Effects of Titanium Additions

The influence of titanium additions of up to 0.6 wt % on the irradiation-induced swelling and microstructural changes, and the unirradiated and post-irradiation creep-rupture properties have been investigated. The salient results are summarized:

- 1) At 500 and 600°C and neutron fluences of 2.0 and $4.4 \times 10^{26} \text{ n/m}^2$, irradiation-induced swelling exhibits a minimum at 0.2 to 0.4 wt % Ti.
- 2) For an alloy having a titanium concentration in this range (specifically 0.23 wt %) the swelling of annealed material is a weaker function of temperature than in standard 316 SS, and the temperature for peak swelling is about 100°C higher.
- 3) At 500 to 600°C, the concentration of voids reached a minimum at 0.2 to 0.4 wt % Ti. The mean void diameter was essentially independent of temperature.
- 4) Cold working the Ti-modified 316 alloy effectively suppressed void formation and swelling at 500 and 600°C and $3.0 \times 10^{26} \text{ n/m}^2$. At $6.6 \times 10^{26} \text{ n/m}^2$, the cold-worked structure exhibited a small amount of swelling at 500°C and no swelling at 600°C.
- 5) In the annealed condition, alloys containing 0.23 and 0.33 wt % Ti exhibited maximum creep-rupture strength. On thermal aging, the strength properties of all alloys decreased. The effect was larger in those alloys containing titanium. After aging for 4000 hr at 650°C, the properties of the standard and 0.23 and 0.33 wt % Ti alloys were about equal.
- 6) The post-irradiation creep-rupture ductility was maximum for alloys containing 0.23 and 0.33 wt % Ti.

The collective influence of elements such as C, N, S, Si, etc., on void nucleation and growth kinetics has been demonstrated.⁽⁵⁾ It can also be concluded from experimental results and indirect evidence that many of these same elements affect both strength properties and ductility or fracture behavior.^(8,9) It is thus not surprising that the addition of a very reactive element such as titanium markedly influences the response to irradiation. As a first step in explaining the observed effects, we will consider the compositional changes that occur in the austenite by the addition of titanium.

It is reasonable to assume that the first additions of titanium remove nickel and sulfur from solution in the austenite as $Ti(C,N)$ and $Ti_4C_2S_2$. Each of these precipitates is present in the alloys containing titanium, and both are stable to very high annealing temperatures.⁽¹⁰⁾ Assuming that all sulfur and nitrogen are removed (along with titanium and carbon) as the above mentioned compounds, the amount of titanium and carbon in solution when equilibrium is obtained at 1050 and 600°C can be estimated. Results are given in Table ORNL-9. For a 1050°C annealing temperature, the amounts of carbon and titanium in solution decrease and increase, respectively, with increasing titanium. At 600°C, assuming equilibrium, the amount of titanium in solution increases from 0 to 0.42 wt % for total titanium contents from 0.23 to 0.60 wt %. Only the alloy containing 0.23 wt % Ti would have any carbon remaining in solution (0.01 wt %). In the other alloys, all carbon would be precipitates as TiC. The general trend for increasing swelling with increasing titanium could be associated with one or more factors: (1) increasing amounts of titanium and decreasing amounts of carbon in solution; (2) increasing amounts of TiC precipitate; or (3) the possibility that the minimum occurs because certain elements such as titanium and carbon in solution retard swelling, while precipitates such as TiC enhance swelling. If equilibrium were attained early in the irradiation, then alloys containing 0.33, 0.46, and 0.60 wt % Ti would have essentially all carbon precipitated and the second possibility could not be controlling. Furthermore, if equilibrium were attained, the amount of TiC precipitate would be essentially constant, and factors 2 and 3 could be eliminated. Whether or not equilibrium was attained has not been established and will be subject to further investigation.

TABLE ORNL-9
COMPOSITION OF SAMPLES COMPUTED FROM CHEMICAL
AND THERMODYNAMIC ANALYSES

	Concentration, wt %				
	R1	S1	T1	U1	DO
Total Ti	0.23	0.33	0.46	0.60	0.05
Ti as $Ti_4C_2S_2$	0.029	0.029	0.029	0.010	0.05
Ti as Ti(CN)	0.029	0.033	0.026	0.026	
C available to react with Ti	0.054	0.044	0.044	0.036	0.044
At 1050°C Equilibrium					
Ti in solution	0.16	0.22	0.32	0.48	0.0
C in solution	0.05	0.03	0.02	0.01	0.04
TiC formed	0.01	0.06	0.11	0.11	
At 600°C Equilibrium					
Ti in solution	0.0	0.09	0.23	0.42	
C in solution	0.01	0.00	0.00	0.00	
TiC formed	0.22	0.22	0.22	0.18	

The precipitates $Ti_4C_2S_2$ and $Ti(C,N)$ appear to be unimportant from the swelling standpoint, because they are usually quite massive and voids are not generally associated with them. The TiC precipitates, on the other hand, are often associated with voids and could be a factor in either void nucleation or growth.

It also appears that increasing silicon content causes the swelling to decrease. The high-purity alloy contained about 0.01% Si, the type 316 SS examined by Brager⁽⁴⁾ about 0.30 wt % Si and heat D0 about 0.75 wt % Si. The swelling decreased with increasing silicon. This is consistent with the recent observations of Brager, et al.⁽¹⁰⁾

The decreased tendency for grain-boundary cracking and fracture in the unirradiated condition appears to be associated with the removal of trace impurities such as sulfur from solution. It is noted that the decreased tendency for intergranular fracture is observed at the lowest titanium level such that all of the sulfur and nitrogen, but not all of the carbon, is removed from solution. Although in the unirradiated condition there was a marked decrease in the amount of grain-boundary cracking in the alloys containing titanium, there was no strong correlation of the total creep elongation. The lack of correlation between grain boundary cracking and total creep elongation in the unirradiated condition probably results from the fact that individual cracks did not undergo extensive propagation. Failure occurred by specimen overload due to reduction of area, rather than by cracks linking together.

Alloys having 0.23 and 0.33 wt % Ti exhibited maximum ductility in post-irradiation creep-rupture tests. A significant difference between these two alloys and the commercial alloy (heat D0) was the fact that at 550°C, the alloys containing the titanium additions exhibited extensive tertiary creep in comparison to little or no tertiary creep in the commercial alloy. The variation in creep strength with titanium is inferred from previous work on titanium additions to 304 SS.⁽¹¹⁾ The increase in rupture life and decrease in minimum creep rate with the initial titanium additions for alloys in the solution treated condition may result from the formation of small precipitates on dislocations and thus a locking of the dislocations during creep deformation. The lower strength properties at still higher titanium levels and the reduction in strength on thermal aging may result from the removal of carbon from solution prior to testing, thus preventing precipitation and the resulting dislocation locking mechanism from operating.

2. Effects of Molybdenum Concentration

The present results show that molybdenum has a marked influence on the temperature-dependence of swelling. In the alloy containing 0.21 wt % Mo, the swelling decreased between 500 and 600°C, while in the alloys containing 2.46 and 5.15 wt % Mo, the swelling increased. The temperature at which the peak swelling occurs may thus have been shifted by over 100°C.

Future work will include TEM examination of samples irradiated to 2.1×10^{26} and 5.6×10^{26} n/m² (>0.1 MeV) to determine the influence of molybdenum on the void concentration and size distributions.

II. EFFECTS OF PRE-IRRADIATION MICROSTRUCTURE ON SWELLING AND MECHANICAL PROPERTIES

E. E. Bloom

Irradiation-induced swelling and loss of ductility are two major problems in the use of 316 SS as a fuel cladding and core structural alloy in LMFBRs. Cold working has been shown to reduce swelling, presumably because the high dislocation density formed during deformation acts as a sink for both vacancies and interstitials. After irradiation in either the cold-worked or annealed condition, this alloy exhibits low creep-rupture ductility because of intergranular fracture. The purpose of this work is to determine whether the post-irradiation ductility can be improved by control of the pre-irradiation microstructure while still maintaining swelling resistance through a high sink density.

A. EXPERIMENT DESCRIPTION

Samples of 316 SS (heat D0, the composition listed in Table ORNL-1) were given various combinations of cold-working and thermal-aging treatment before irradiation in EBR-II subassembly X100 and X100A. The bulk of the samples were irradiated at 500 and 600°C although for some heat treatment conditions, irradiation temperature of 400, 450, and 700°C were also included. The maximum fluence was $7.8 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$).

B. RESULTS

Results of immersion density measurements, transmission electron microscopy (TEM), and post-irradiation creep-rupture tests on samples irradiated in the previous report.⁽²⁾ During the present reporting period, density measurements, transmission microscopy, and creep-rupture tests on samples from subassembly X100A were initiated.

1. Immersion Density Changes

Immersion density changes in 316 SS (heat D0) irradiated in the annealed and 20% CW conditions at 500 and 600°C are listed in Table ORNL-10. It is particularly significant that this heat of 316 SS exhibits very low swelling, even in the annealed condition. Swelling correlations of Brager and Straalsund⁽⁹⁾ predict density decreases of about 5.0 and 2.4% in annealed 316 SS irradiated to $6.6 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$) at 500 and 600°C compared to the values of 0.59 and 1.53% observed in this alloy. Heat D0 also exhibits lower swelling than the alloy having nominally the same composition (heat 02) described in the previous section (heat 02), each exhibit higher swelling at 600 than at 500°C, where the opposite is predicted from the swelling correlation. This may arise from the fact that the correlation was based on the available data that extended up to about $3 \text{ to } 4 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$). It appears that the temperature for maximum swelling increases with fluence.

It is possible that heat D0 simply has a longer incubation period before the onset of swelling. If the responsible factors can be determined, they can then be used and optimized in the development of swelling resistant "300 series" stainless steels where swelling resistance implies acceptable swelling behavior over the range of temperature and fluence of interest in the design of LMFBRs. The required swelling resistance could result from either an extended incubation period or lower swelling rate after the onset of swelling.

TABLE ORNL-10
RADIATION-INDUCED DENSITY CHANGES IN ANNEALED
AND 20% CW 316 SS^a

Irradiation Temperature (°C)	Fluence, >0.1 MeV (n/m ²)	Density Decrease, %	
		Annealed	20% Cold Worked
500	5.6×10^{26}	0.37	-0.10
	6.6	0.59	
600	5.6	0.31	0.40
	6.6	1.53	

^aSamples from heat DO, composition listed in Table ORNL-1.

Results of immersion density measurements on samples irradiated in other heat treatment conditions are listed in Table ORNL-11.

TABLE ORNL-11
EFFECT OF HEAT TREATMENT ON DENSITY CHANGES
IN IRRADIATED 316 SS^a

Heat Treatment	Fluence, >0.1 MeV (n/m ²)	Density Change, % At			
		450°C	500°C	600°C	700°C
1 hr 1050°C + 100 hr at 700°C + 20% CW	7.8×10^{26}	+0.05	+0.13	-0.71	+0.24
1 hr 1050°C + 20% CW + 1 hr at 700°C	7.8		-0.35	-0.45	
1 hr 1050°C + 20% CW + 10 hr at 650°C	7.8	-0.35	-0.48 ^b	+0.08 ^b	-0.06
1 hr 1050°C + 20% CW + 10 hr at 550°C	6.6		-0.10	-0.05	

^aIrradiated to 7.8×10^{26} n/m² (>0.1 MeV).

^bFluence on samples was 6.6×10^{26} n/m² (>0.1 MeV) data normalized to 7.8×10^{26} n/m² (>0.1 MeV) assuming $\frac{\Delta\rho}{\rho} \propto (\phi t)$.

All of the samples having alternate pre-irradiation heat treatments exhibited low swelling similar to the behavior of samples that were irradiated in the annealed or 20% CW conditions. It does appear that the heat treatments may have shifted the temperature-dependence of the swelling (e.g., annealing 1 hr at 1050°C followed by aging 100 hr at 700°C and then cold working 20% resulted in maximum swelling at 600°C compared to annealing 1 hr at 1050°C followed by cold working 20% and aging 10 hr at 650°C, which resulted in maximum swelling in the neighborhood of 500°C). Samples that were annealed 1 hr at 1050°C then cold worked 20% and aged 10 hr at 550°C exhibited the lowest swelling when the results of both 500 and 600°C irradiations are considered.

Post-irradiation creep-rupture tests on samples irradiated in subassembly X100A were initiated in the last reporting period. Since not all tests in the series have been completed, it is difficult to make general conclusions. Results for which comparisons can be made at two different fluence levels are listed in Table ORNL-12.

The rupture life of samples irradiated in the annealed and annealed + 20% CW + 10 hr at 550°C conditions decreased by a factor of 3 to 3.5 when the fluence was increased from 3.0×10^{26}

n/m^2 (>0.1 MeV). This further decrease of rupture life was attributable to an increase in creep rate. The sample that was annealed then CW 20% and aged 10 hr at 650°C exhibited little change in creep-rupture properties with the increase in fluence. This may be associated with the relative stability of this structure at the 500°C irradiation temperature.

TABLE ORNL-12
EFFECT OF PRE-IRRADIATION HEAT TREATMENT ON POST-IRRADIATION
CREEP-RUPTURE PROPERTIES OF 316 SS

Preirradiation Heat Treatment	Samples Irradiated to					
	$3.0 \times 10^{26} \text{ n/m}^2$ (>0.1 MeV)			$6.6 \times 10^{26} \text{ n/m}^2$ (>0.1 MeV)		
	Rupture Life (hr)	Creep Rate (%/hr)	Creep Elongation (%)	Rupture Life (hr)	Creep Rate (%/hr)	Creep Elongation (%)
Irradiated at 500°C, Tested at 550°C 55 ksi (379.2 MPa)						
1 hr at 1050°C	161	7.4×10^{-3}	1.8	60	4.4×10^{-2}	3.3
1 hr at 550°C + 20% CW + 10 hr at 550°C	1260	2.4×10^{-3}	5.7	365	1.0×10^{-2}	5.3
1 hr at 550°C + 20% CW + 10 hr at 650°C	549	5.8×10^{-3}	4.8	700	7.2×10^{-3}	8.4
Irradiated at 600°C, Tested at 650°C 25 ksi (172.3 MPa)						
1 hr at 1050°C	458	1.4×10^{-2}	15.6			
1 hr at 1050°C + 20% CW + 10 hr at 550°C	114	3.8×10^{-2}	7.8	24	1.6×10^{-1}	4.7
1 hr at 1050°C + 20% CW + 10 hr at 650°C	111	3.2×10^{-2}	6.7	22	1.4×10^{-1}	3.6

The samples that were annealed, cold worked 20% and then aged 10 hr at 550 or 650°C exhibited a factor of 5 decrease in rupture life when irradiated at 600 and tested at 650°C and 25 ksi (172.3 MPa). Similar to the samples irradiated to $3.0 \times 10^{26} \text{ n/m}^2$ (>0.1 MeV), the post-irradiation properties remained nearly identical at the higher fluence. This apparently resulted from instability of the microstructure of the sample that was cold worked and then aged 10 hr at 550°C leading to an approach of a common microstructure for the two heat treatments.

C. FURTHER WORK

Samples representing each of the preirradiation heat treatments have been cut. These samples will be examined by transmission electron microscopy to relate properties and property changes to microstructure and microstructural changes. A number of creep-rupture tests on samples having other preirradiation heat treatments remain to be completed.

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III. CONTROLLED THERMONUCLEAR REACTOR PROGRAM--EFFECTS OF HIGH-HELIUM-GENERATION RATES ON RADIATION DAMAGE IN STAINLESS STEELS*

F. W. Wiffen and E. E. Bloom

These investigations are devised to determine the effects of high-helium-production rates on radiation damage (swelling kinetics, microstructure, and mechanical property changes) in austenitic stainless steels and nickel-base alloys. These materials have been suggested as possible first-wall construction materials for CTR's.** At a neutronic wall loading of 1.87 MW/m², approximately 30 displacements per atom (dpa) and 460 ppm (atomic) He will be produced in stainless steel per year of CTR operation.

In the High Flux Isotope Reactor (HFIR), helium is produced by a two-step nuclear reaction with thermal neutrons: $^{58}\text{Ni}(n,\gamma)^{59}\text{Ni}$ followed by $^{59}\text{Ni}(n,\alpha)^{56}\text{Fe}$, with effective cross sections of 4.2 and 7.4 barns, respectively. In alloys containing nickel, the helium concentration has been found experimentally to increase as $(\phi t)^{1.68}$ (Ref. 3), and irradiation of stainless steel in HFIR results in about 60 dpa and 1900 ppm He in the first year. The neutron spectrum and flux available in HFIR thus allow us to approximate displacement and helium production rates expected in a stainless steel CTR first wall.

Specimens irradiated in HFIR are being examined to determine the amount of irradiation-induced swelling and the effects of irradiation on the microstructure, tensile, creep, and fracture behavior. When property measurements are completed, the results will be related to the potential use of stainless steels in proposed CTR environments.

*Work supported by the AEC Division of Controlled Thermonuclear Research.

**Work conducted at Plasma Physics Laboratory, Princeton, N.J. by P. Bonanos and W. Price, Jr. Also, see Reference 2.

A. PREVIOUSLY REPORTED RESULTS

Annealed and 20% CW specimens of standard 316 SS were irradiated in HFIR at temperatures between 370 and 785°C to fluences $\leq 8.7 \times 10^{26}$ n/m² (>0.1 MeV). Details of specimen chemistry and processing and of the irradiation experimental methods and irradiation parameters were reported previously.⁽⁴⁾

Results of swelling determined by immersion density measurement and TEM on one sample, presented previously,⁽⁴⁾ showed the matrix to contain a high concentration of cavities assumed to be helium-containing bubbles. Observations drawn from comparison of the measured swelling and swelling calculated from models showed:

- 1) In solution-treated samples, the swelling is appreciably greater than predicted from displacement damage (EBR-II results) and is somewhat less than predicted for equilibrium helium bubble swelling.
- 2) Cold work is effective in suppressing swelling in the presence of these high helium contents for irradiation temperatures of 600°C and lower but is ineffective at 680°C, probably due to recovery of the cold-worked microstructure.
- 3) The temperature-dependence of swelling is quite unlike that observed in fast reactor irradiations with low helium contents. The swelling is only weakly temperature-dependent between 380 and 600°C for both solution-treated and CW samples and increases slightly in annealed and rapidly in CW samples at 680°C.

Post-irradiation tensile and creep-rupture test results were also reported previously.⁽⁴⁾ Samples irradiated in the annealed condition exhibited an increase in yield strength between 350 and about 600°C. Except at 350°C, the ultimate tensile strength at all temperatures was less than the unirradiated values. The material exhibited little strain hardening, and the total elongation was reduced to 1% over the entire temperature range. Samples irradiated in the 20% CW condition exhibited a decrease in both yield and ultimate tensile stress at all temperatures. The total elongation was only slightly affected by irradiation at 350°C (reduced from 5 to about 4%). However, with increasing irradiation and test temperature, the elongation decreased, becoming zero at 650°C. Post-irradiation creep-rupture tests of both the annealed and CW structures exhibited large reductions in rupture life and ductility due to irradiation.

B. OPTICAL METALLOGRAPHY

Samples of both annealed and 20% CW material irradiated at 380 and 680 to 685°C and tensile tested near the irradiation temperature were examined by optical metallography. Specimens were prepared in a hot-cell metallography line and were etched with aqua regia. Irradiation parameters and tensile test results for the samples examined are given in Table ORNL-13 which summarizes results presented in more detail previously.⁽⁴⁾

Optical metallography showed that extensive precipitation had occurred for all irradiation conditions studied. Photomicrographs from various positions along the gage length of samples irradiated in the annealed condition at 380°C (for 16,200 hr) and 685°C (for 12,800 hr) are shown in Figures ORNL-6 and 7. At each temperature, the amount of precipitates was less in the shoulder than in the vicinity of the fracture as a result of the temperature gradient that existed during irradiation. At 380°C (Figure ORNL-6), a very fine precipitate was present in matrix of the fracture region, but not the shoulder. Intragranular precipitation would not

occur as a result of thermal aging for 16,200 hr at 380°C on the basis of results of Wiess and Stickler⁽⁵⁾ and Spruiell et al.⁽⁶⁾ The present results thus indicate an enhancement of the precipitation kinetics (possibly formation of $M_{23}C_6$) during irradiation, in agreement with previous observations of Brager, et al.⁽⁷⁾ After irradiation for 12,800 hr at 685°C, a number of phases such as $M_{23}C_6$, sigma, chi, and Laues could be present. The lightly etched, massive grain boundary phase (Figure ORNL-7) is either sigma or chi.⁽⁵⁾ One of the intergranular phases is probably $M_{23}C_6$, but other phases could be present as well.

TABLE ORNL-13
IRRADIATION AND TENSILE TEST PARAMETERS FOR 316 SS
OPTICAL METALLOGRAPHY SAMPLES^a

Irradiation Temperature (°C)	Fluence, >0.1 MeV (n/m ²)	Displacements per Atom (dpa)	Helium (ppm)	Tensile Test Temperature (°C)	Stress		Total Elongation (%)
					Yield [ksi (MPa)]	Ultimate [ksi (MPa)]	
Annealed							
380	7.1 × 10 ²⁶	97	4020	350	107.5(741)	109.5(755)	0.61
685	6.6	90	4300	650	25.9(179)	25.9(179)	0.2
20% Cold Worked							
380	7.1	97	4020	350	109(752)	115.8(798)	3.8
680	8.7	121	6090	650	12.7(87.6)	12.7(87.6)	0

^aFurther details were given in *Radiation Effects Program Semiannual. Progr. Rep. Jan. 31, 1974*, ORNL-TM-4500, p. 21.

Irradiation of this material in the 20% CW condition at 380°C (Figure ORNL-8a) resulted in precipitation on the deformation bands that were formed during the cold working prior to irradiation. As in the case of the annealed condition, such precipitation would not be expected from the thermal history alone. At 685°C, the amount of light etching grain-boundary phase (presumably sigma) was considerably greater than found in the samples irradiated in the annealed condition. Other precipitates, often aligned, were present in the matrix, suggesting that they may have formed on the deformation bands. Many of the grain boundaries contained still another structure in addition to the precipitates that have been tentatively identified as sigma. This structure was attacked vigorously by the HCl-HNO₃ etchant normally used for this alloy. Re-polishing and etching lightly revealed regions containing small precipitates shown in Figure ORNL-8b). The identity of this feature is presently unknown. It formed primarily at grain boundaries. The sigma phase particles present in this phase were often rounded and smaller than the particles that extend along grain boundaries in regions where this unidentified phase (or phases) did not form. This may be an indication that the sigma phase was being dissolved.

Photomicrographs of the fracture regions of samples irradiated in the annealed condition at 380 and 685°C and tensile tested at 350 and 650°C are shown in Figures ORNL-8 and 9. At the lower temperature (Figure ORNL-8), the fracture was predominately transgranular. The sample exhibited very little necking or reduction of area and only the very outer rim or edge [about 0.005 in. (0.127 mm)] appeared to fail in shear. At 650°C, Figure ORNL-10, the fracture was

FIGURE ORNL-6. Photomicrographs from Various Positions Along the Length of an Annealed 316 SS Sample that was Irradiated at 380°C and Tensile Tested at 350°C. The different etching behavior apparently results from different amounts of carbide precipitation due to a temperature gradient along the sample. (a) Fracture, (b) midway between fracture and shoulder, and (c) shoulder. All micrographs 100x.

FIGURE ORNL-7. Photomicrographs from Various Positions Along the Length of an Annealed 316 SS Sample that was Irradiated at 685°C and Tensile Tested at 650°C. The lightly etched grain boundary phase is believed to be sigma. The amount decreases from the fracture to the shoulder due to the temperature gradient during irradiation. (a) Fracture, (b) midway between fracture and shoulder, and (c) shoulder. All micrographs 500x.

FIGURE ORNL-8. Microstructures of 316 SS Irradiated in the 20% CW Condition; Left: 500X; Right: 100X; Irradiated at (a) 380°C and (b) 685°C. Photomicrographs were taken from positions midway between the fracture and the shoulder.

FIGURE ORNL-9. Photomicrographs of the Fracture of Annealed 316 SS Irradiated at 380°C and Tensile Tested at 350°C. 100X.

FIGURE ORNL-10. Photomicrographs of Fracture Region of SA 316 SS Irradiated at 685°C in HFIR and Tensile Tested at 650°C. (a) 500x and (b) 100x.

clearly intergranular. Cracks were always observed at the sigma phase-austenite interface and never through the sigma phase particles.

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RADIATION EFFECTS ON METALS

J. L. Brimhall, E. P. Simonen and H. E. Kissinger

I. INTRODUCTION

Evans has recently published results on ion bombardment of single crystal molybdenum showing very low swelling values at 900°C.⁽¹⁾ In a recent paper, the present authors obtained greater swelling values in 7.5 MeV Ta ion-bombarded, single crystal molybdenum for a similar temperature range.⁽²⁾ Due to experimental malfunctions, the actual temperature was not known precisely, but was estimated to be about 940°C (± 400). Some of the experiments were rerun and the temperature range expanded to 800°C to 1000°C, and these results are presented in this note. The void parameters and swelling are compared with those reported by Evans. Some differences are still apparent and some possible reasons for the differences are discussed.

II. EXPERIMENTAL

The experimental procedure was similar to that reported in Reference 2, with some exceptions. The displacement damage as a function of ion penetration was determined from the computer code of Manning and Mueller.⁽³⁾ This gives slightly larger displacement values than that given by the previously used calculation of Kulcinski, et al.⁽⁴⁾ The depth distribution of the damage is shown in Figure PNL-1.

The temperature of bombardment was 800°C, 900°C, and 1000°C, with most data obtained at 1000°C. Because of thermocouple malfunction experienced in the work of Reference 2, additional care was taken to insure that the specimen was in fact at the desired temperature. In this work, the temperature of the furnace was calculated from optical pyrometer readings and the control thermocouples calibrated to these readings. The estimated accuracy of the readings is $\pm 30^\circ\text{C}$. The temperature variation during a run was less than $\pm 10^\circ\text{C}$. The void lattice parameter is known to be a function of reciprocal temperature and was used as an additional index of irradiation temperature.

Some specimens did receive some post-irradiation annealing at the bombardment temperature and the time of annealing extended up to five hours for some samples.

III. RESULTS AND DISCUSSION

Most of the data was obtained at 1000°C. Only two high dose specimens were run at 900°C to check previous results. At 800°C, the voids were very small, even at the highest dose, and a density could not be accurately calculated. There was also a high density of small dislocation loops which hindered void identification at 800°C bombardment. A comparison of the void parameters for the three temperatures and similar doses is given in Table PNL-1. The void density at 1000°C computed from the lattice parameter of 460 Å is 2.1×10^{16} voids/cm³. The actual density measured varied between 1.4 and 1.7×10^{16} voids/cm³. The fraction of sites occupied in the lattice therefore varies from 65 to 80%. There were void-free islands

FIGURE PNL-1. Displacement Damage in Molybdenum as a Function of Distance from the Bombarded Surface after Bombardment with 7.5 MeV Ta⁺⁺⁺ Ions to a Dose of 3×10^{16} Ions/cm².

within the void lattice, which principally accounts for the lower calculated void density. Similar observations of the void lattice have been made after the 900°C irradiations. The void lattice is not observed in the 800°C irradiations.

TABLE PNL-1
VOID PARAMETERS FOR THREE ION
BOMBARDMENT TEMPERATURES

Temperature (°C)	Dose (dpa)	Size (Å)	Density (cm ⁻³)	$\Delta V/V$	Void Lattice Parameter (Å)
800	150	~25	$>10^{17}$	1%	---
900	170	76	4.5×10^{16}	2.5%	320
1000	160	105	1.5×10^{16}	2.2%	460

The void diameter is plotted as a function of dose at 1000°C in Figure PNL-2. Data from the previous work is also plotted.⁽²⁾ The volume averaged size $\bar{d} = \left(\frac{\sum n_i d_i^3}{\sum n_i} \right)^{1/3}$ is used in Figure PNL-2. These sizes are larger than the arithmetic average by 5 to 10%. The voids are generally cubic and the cube edge is used as a size measure. Truncation of the voids is prevalent in the larger voids, and this truncation is taken into account in computing void volume fraction.

FIGURE PNL-2. Void Size (Cube Edge) as a Function of Number of Displacements for Molybdenum Bombarded at Approximately 1000°C with 7.5 MeV Ta⁺⁺⁺ Ions.

The calculated void volume fraction ($\Delta V/V$) is plotted as a function of dose in Figure PNL-3. For comparison purposes, the data from Evans' work at 900°C is also shown. A straight line is drawn through the points with a slope of approximately 2/3. The swelling reaches a value of 2 to 3% at the highest dose of 150 dpa. There is no clear indication of saturation at this point. Evans' swelling values are much lower, but it is possible to draw a similar slope through the data points.

The results in Figure PNL-3 indicate that the swelling in molybdenum is significantly greater at 1000°C than at 900°C, even though the dose-dependence of the swelling may be the same. However, the limited data at approximately 900°C given in Table PNL-1 still shows swelling values of 2.5%, which is a factor of 8 higher than that shown in Figure PNL-3 from Evans' work. The discrepancy between the present results and those reported by Evans must be due to inconsistencies in experimental procedure, irradiation parameters or material condition. In the results presented in this note, a cube shape is assumed in the void volume calculation, as opposed to a spherical shape assumed by Evans. This difference in experimental procedure

FIGURE PNL-3. Void Swelling as a Function of Number of Displacements for Molybdenum Bombarded with 7.5 MeV Ta⁺⁺⁺ Ions at 1000°C and 2 MeV N⁺ Ions at 900°C (Evans' Data).

may account for a factor of two in swelling. Although the specimens in the present study experienced post-bombardment annealing at the irradiation temperature, this difference in sample history can not account for an increase in swelling. Even allowing for these procedural differences, there is still considerable disagreement in the swelling values which must be due to irradiation or material effects.

The irradiation temperature is a point of concern in the present work. It is possible that the expected temperature was not in each case realized during the irradiation. To investigate this possibility, the measured void lattice parameters from the present work, the previous work and some earlier work of Evans were compared. Figure PNL-4 illustrates the relationship between the intended irradiation temperature and the measured void lattice parameter. A straight line has been drawn through the 1000°C data point of this work and the 870°C data point of Evans. This has been done since these two temperatures are known with the greatest confidence. It is seen that the two data points at 900°C are in disagreement with those at 870°C and 1000°C. For this reason, it is believed that the actual irradiation temperature in the previous work was closer to 1000°C and that in the present study the intended 900°C irradiation was closer to 925°C. The proposed temperature correction is shown by the horizontal arrow in Figure PNL-4. Thus, a strong temperature-dependence of swelling near 900°C would in part rationalize the disparity in observed swelling between the present work and that of Evans.

FIGURE PNL-4. Measured Void Lattice Parameter as a Function of Reciprocal Temperature from this Study and References (s) and (5). The Horizontal Arrows Indicate the Predicted Temperature Corrections.

The principal difference in material condition between the specimens of this study and those of Evans is the presence of 10 ppm He in the latter investigation. The higher void density observed by Evans is consistent with helium-enhanced void nucleation. Copious void nucleation can result in an imbalance between the efficiency of interstitial type sinks and vacancy type sinks. Such a situation could contribute to the lesser swelling observed by Evans.

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RADIATION EFFECTS ON THE BCC METALS AND ALLOYS

John Moteff and James Stubbins

I. OBJECTIVE

An extensive study of substructure and physical properties of neutron- and ion-irradiated BCC metals and alloys is being carried on in order that insight in their performance in operating reactor environments may be gained. Physical and mechanical properties of irradiated metals are related directly to substructure. Substructural investigations of as-irradiated, and irradiated and annealed material are used to determine the nature and thermal stability of defects and to correlate the presence of defects with changes in measured physical properties. The influence of several variables on the radiation damage structure of the BCC metals are being studied as an integral part of this program. The irradiation variables include the effects of dose, dose rate, temperature and irradiating particle spectrum on the nature and distribution of defects. The material variables include the effects of impurity atoms and substitutional alloying atoms. The effect of post-irradiation annealing of irradiated material is also investigated.

The materials being studied in this program include several BCC metals and their alloys: Mo: Mo-0.5Ti; TZM; Nb; Nb-1Zr; W; and W-25Re. The irradiation conditions are mentioned where appropriate in the following test. The nature and thermal stability of irradiation induced defects are studied using transmission electron microscopy (TEM), length change measurements, electrical resistivity measurements, bulk density measurements, and microhardness measurements. The progress in several phases of this program are reported below.

II. RADIATION EFFECTS ON Mo AND Mo ALLOYS

A. DIMENSIONAL STABILITY OF NEUTRON-IRRADIATED MOLYBDENUM

H. Jang

It is important for reactor design considerations to be able to predict the dimensional changes of a metal and/or alloy for the various combinations of operating temperatures, neutron fluences, neutron flux densities and spectra and stress levels. The thermal stability of any irradiation induced length change is also needed. In light of this, the results of a study of polycrystalline molybdenum specimens irradiated in the ORR and the EBR-II facilities at temperatures between 70° and 480°C to fast neutron fluences in the range of 10^{19} to 10^{22} n/cm² (E > 1 MeV) are presented here.

Changes in linear dimensions of molybdenum following fast neutron irradiation were measured using a high vacuum dilatometer which is described in detail elsewhere. Specimens were prepared from commercially pure (99.98%) polycrystalline molybdenum in the form of 3.17 mm and 1.27 mm diameter rods, about 50 mm long. The principal impurity of the specimens was 37 ppm of carbon. The large diameter (3.17 mm) specimens were irradiated in the Oak Ridge Research

Reactor (ORR) or Experimental Test Reactor (ETR) to fast neutron fluences ranging from 1.1×10^{19} n/cm² to 1.5×10^{21} n/cm² ($E \geq 1$ MeV) at three different temperatures, 70°C, 380°C and 480°C. The small diameter (1.27 mm) specimens were irradiated in the EBR-II to a fast neutron fluence of 1×10^{22} n/cm² at 430°C. The irradiation conditions of each specimen are tabulated in Table UC-1.

The irradiated specimens were then annealed inside the furnace of the high vacuum dilatometer. The irradiated specimens were heated continuously with a linear heating rate of 0.5°C/min. from 70°C to 900°C, and then held at the highest temperature for two hours before cooling. The temperature and length changes of the specimens were continuously plotted on an X-Y recorder, and then the specimens were isochronal-annealed in another vacuum furnace system for one hour at each additional annealing temperature from 1000°C to 1600°C.

Total fractional length change, $\Delta l/l$, was calculated by subtracting the length of the post-irradiation annealed specimen from that of the as-irradiated specimen as follows:

$$\frac{\Delta l}{l} = \frac{l_i - l}{l}$$

where l_i = length of as-irradiated specimen
 l = length of post-irradiation annealed specimen.

The accuracy of the dilatometric measurement was within 0.001 percent for 50-mm specimens.

All the specimens examined in this study showed an increase in length after neutron irradiation. At an irradiation temperature of 70°C, the irradiation-included length changes increased with the fast neutron fluences. The rate of length changes increased with the fluence approaching a saturation level of 0.05 ~ 0.06 percent at a fluence of $\sim 10^{20}$ n/cm² or higher. Length changes of the specimens irradiated at 380°C and 480°C to a neutron fluence of 5×10^{19} n/cm² decreased with increasing irradiation temperature up to 500°C with a minimum occurring at 500° and 600°C.

The specimen irradiated in the EBR-II at 430°C to a neutron fluence of 1×10^{22} n/cm² showed a length change of 0.325 percent, which is about 15 times larger than those irradiated in the ORR. Transmission electron microscopy on this specimen revealed the presence of voids. A summary of the irradiation-induced length changes of all the specimens examined in this study is given in Table UC-1.

Upon annealing, the irradiation-induced length changes recovered. A complete recovery of the ORR specimens occurred at $\sim 800^\circ\text{C}$, and that of the EBR-II occurred at $\sim 1300^\circ\text{C}$. The recovery curves of the ORR specimens are shown in Figure UC-1. These specimens show four recovery stages at $0.16 T_m$ (Stage III), $0.22 T_m$ (Stage IV), $0.31 T_m$ (Stage V), and $0.36 T_m$ (Stage VI), respectively. The recovery curve of the EBR-II specimen is shown in Figure UC-2. This specimen shows only two recovery stages at $0.36 T_m$ (Stage VI) and $0.54 T_m$ (Stage VII).

When an equal number of single interstitial atoms and vacancies are introduced in a crystal, the volume of the crystal changes due to the lattice relaxation around point defects. If one or both types of point defects are mobile, then the mobile species will: 1) form clusters by interaction between like-defects; 2) anneal out by migrating to free surfaces; or 3) be annihilated by interaction between unlike-defects. The length of a crystal always increases regardless of the type of concentration of point defects introduced. The effects

of irradiation temperature and neutron fluence on the microstructure developed in molybdenum have been well characterized by TEM in a number of investigations.^(1,2) The length change data are categorized into three groups according to irradiation temperature and neutron fluence.

TABLE UC-1

IRRADIATION-INDUCED LENGTH CHANGES OF MOLYBDENUM AND RECOVERY PEAK TEMPERATURES

Mo Spec No	Irradiation Condition		Sample Dimension (mm)	Total Length Change (%)	Peak Temperature of Recovery Spectra				
	Fluence, ϕ (n/cm^2)	Temp. ($T_i, ^\circ C$)			Stage III 0.16 T_m	Stage IV 0.22 T_m	Stage V 0.31 T_m	Stage VI 0.36 T_m	Stage VII 0.54 T_m
504(a)	1.1×10^{19}	70	51.0x3.0	0.023	175°C	320°C	635°C	785°C	N.O. ^(c)
2284(a)	7×10^{19}	70	47.6x3.0	0.041	180°C	320°C	635°C	770°C	N.O.
925(a)	1.5×10^{21}	70	50.7x3.0	0.056	175°C	420°C	640°C	760°C	N.O.
2275(a)	5×10^{19}	380	47.6x3.0	0.020	N.O.	N.O.	655°C	800°C	N.O.
2285(a)	5×10^{19}	480	47.6x3.0	0.018	180°C	N.O.	695°C	775°C	N.O.
1A-4(b)	1.10^{22}	430	47.6x1.3	0.325	N.O.	N.O.	N.O.	795°C	1,280°C

(a) Other specimens irradiated in ORR.

(b) Irradiated in EBR-II.

(c) N.O. - Not Observed.

In the low temperature ($\sim 70^\circ C$) - low fluence (up to $10^{21} n/cm^2$) region, the damage structure consists mainly of black spots, small dislocation loops and rafts. These cluster defects are believed to be predominantly of the interstitial type^(2,3,4), suggesting that one type of interstitial defect is mobile at, or below reactor ambient temperature in a reactor environment. Others have suggested that interstitials are mobile below room temperature in molybdenum.⁽⁵⁾ The length changes in this temperature region are mainly due to the accumulation of isolated vacancies.

TEM micrographs of the specimens post-irradiation annealed at $550^\circ C$ show almost no change in cluster defect size and density, but on annealing between $700^\circ C$ and $830^\circ C$, the loop size increases significantly with a large decrease in loop density.⁽⁴⁾ Hence, the recovery Stage III and IV observed in the length change measurements are likely to be due to the annealing of point defects, and the annealing of cluster defects is responsible for the recovery Stages V and VI. The ratio of the amount of length recovered in Stages III and IV to that in Stages V and VI becomes smaller with the increasing fluence, suggesting that the ratio of point defects to cluster defects decreases with the fluence.

In the high temperature ($380^\circ C$ - $480^\circ C$) - low fluence ($5 \times 10^{19} n/cm^2$) region the as-irradiated microstructures are similar to those observed at $\sim 70^\circ C$. However, the defect structures are coarsened, and the regions along the grain boundaries are free of defects (grain boundary denuded zone). The quenched-in vacancies are believed to be migrating in this temperature region.⁽⁶⁾ In this case, the vacancies could have formed voids which would have resulted in large length changes. However, the vacancies were observed in the form of dislocation loops^(4,7)

and no voids were observed. This is consistent with the small length changes observed in these specimens. The recovery Stages III and IV were not observed in the specimen irradiated at 380°C suggesting that only cluster defects were remaining in the as-irradiated specimen. However, the specimen irradiated at 480°C did reveal the recovery Stage III.

FIGURE UC-1. Recovery of Fast Neutron Irradiation-Induced Length Increment of Recrystallized Molybdenum as a Function of Annealing Temperature.

The high temperature (430° to 1000°C) - high fluence (10^{22} n/cm^2) region is characterized predominantly by void formation. The swelling increases slightly with the irradiation temperature, reaching a maximum at $\sim 600^\circ\text{C}$. In this temperature region, both the size and concentration of the voids remain constant. In the temperature region between 600° and 800°C, the voids start growing slowly while the concentration decreases, resulting in a net decrease in swelling. At temperatures higher than 800°C, the voids grow rapidly and swelling again increases.

FIGURE UC-2. Recovery of Fast Neutron Irradiation-Induced Length Increment of Recrystallized Molybdenum as a Function of Annealing Temperature.

Upon post-irradiation annealing, two recovery regions were observed. The voids, observed in as-irradiated and post-irradiation annealed specimens at 900°C, were completely annealed out after 1600°C annealing. Hence, the observed recovery stage at $0.54 T_m$ is believed to be due to the annealing of voids. A TEM study⁽⁴⁾ also confirmed the annealing of voids in this temperature region and no void was observed in molybdenum after neutron irradiation at $\sim 1380^\circ\text{C}$.⁽⁸⁾

B. ION BOMBARDMENT OF MOLYBDENUM AND MOLYBDENUM ALLOYS

J. Stubbins

Specimens of Mo, Mo-0.5Ti, and Mo-TZM were irradiated with 3.1 MeV vanadium ions to several doses up to 30 dpa at 800°, 900°, 950° and 1050°C. These irradiations were performed at Argonne National Laboratory using the Dynamatron Facility. A study to determine the temperature and dose dependence of swelling and defect production is now in progress. This study will complement a study of neutron irradiated Mo and Mo-0.5Ti⁽⁴⁾ which has been completed. The neutron irradiated Mo and Mo-0.5Ti specimens received a dose of ~ 21 dpa ($\sim 1 \times 10^{22}$ n/cm², $E_n > 1$ MeV in EBR-II Row 7 midplane) and were irradiated at six different temperatures (approximately 430, 580, 700, 800, 900 and 1050°C), thus a comparison with the results from the ion irradiated specimens will be possible.

The disparities in presently published studies of ion bombarded Mo^(9,10) indicate that this further study should help to clarify some of the uncertainties as to the effect of irradiating temperature and alloying additions on void swelling behavior. An indication of the effect of the choice of irradiating species may also possibly become apparent.

III. RADIATION EFFECTS ON W AND W-25Re

A. SWELLING IN NEUTRON IRRADIATED TUNGSTEN AND TUNGSTEN-25% RHENIUM

J. Matolich and H. Nahm

Some recent observations on the swelling of irradiated tungsten and tungsten-25% rhenium specimens which were irradiated at six different temperatures to a fast neutron fluence of about 1×10^{22} n/cm² ($E > 1$ MeV), are presented here. The data were generated as part of a study⁽¹¹⁾ on the effects of neutron irradiation on the BCC metals and alloys.

Tungsten specimens used in this work were 1 mm in diameter by 70 mm long fabricated from commercially pure, 99.98%, sintered, and swaged tungsten stock and given a pre-irradiation recrystallization heat treatment at 1750°C for one hour in a vacuum environment. The tungsten-25 rhenium specimens were 2 mm in diameter with less than 30 ppm interstitial impurities and had been centerless ground from vacuum arc melted, extruded, hot swagged material followed by a recrystallization heat treatment.

All specimens had been irradiated in Row 7 of the Enriched Breeder Reactor (EBR-II) to a fast neutron fluence of $\sim 1 \times 10^{22}$ n/cm² ($E > 1$ MeV) or about 5.5×10^{22} n/cm², ($E > 0.1$ MeV). The fast neutron fluence has been calculated from the neutron spectrum presented by J. L. Kamphouse, et al.⁽¹²⁾. The fast neutron flux density for $E > 0.1$ MeV and $E > 1$ MeV were 2.3×10^{13} and 4.2×10^{12} n/cm² MW⁻¹ sec⁻¹, respectively.

The irradiation temperatures were determined by design calculations and were checked by measurements of the lattice expansion of silicon carbide detectors placed near the specimens and compared with the available data on the lattice expansion of silicon carbide as a function of

irradiation temperature and fluence. Uncertainties in the first five temperatures were determined to be $430 \pm 5^\circ$, $580 \pm 20^\circ$, $680 \pm 30^\circ$, $800 \pm 20^\circ$, and $900 \pm 20^\circ$. Work in progress will give the uncertainty for the one upper temperature, which is believed to be about 10% higher than the design temperature of 1000°C .

Several aspects of the swelling of these two irradiated materials as measured by swelling recovery, are given in Figures UC-3 through UC-5. Both immersion density and dimensional measurements were used to determine the degree of swelling.

FIGURE UC-3. Observed Swelling of Irradiated Tungsten (○) and Tungsten-25 Rhenium (□). Based on Immersion Density Measurements.

FIGURE UC-4. Observed Swelling of Irradiated Tungsten. Based on Length (■) and Diameter (▲) Dimensional Changes.

Figure UC-3 shows the results of immersion density determinations on the two materials. These data were measured in an apparatus⁽¹³⁾ based on a technique of a direct density comparison⁽¹⁴⁾ with the unirradiated control specimen. It can be seen that the addition of 25 percent rhenium to tungsten suppresses the swelling in the temperature range covered. The lack of swelling is supported by transmission electron microscopy. No voids have been observed in the W-25Re alloy. However, a large fraction of second phase precipitate was observed⁽¹⁵⁾, although this composition is normally single phase.

Radiation-caused atom displacements can provide the sites for precipitation within the collision cascade region. Irradiation-induced precipitation appears to suppress the swelling by disturbing the proper conditions for void formation. In particular, the supersaturation of vacancies needed for the formation of voids can be used for enhancing precipitation of second phase during irradiation, and, subsequently, the interfaces of the precipitates can also form convenient sites for recombination of vacancies and interstitials.⁽¹⁶⁾ However, the exact

mechanism of this behavior is not known. The experimental data clearly demonstrates that certain compositions, similar to the V-20Ti alloys⁽¹⁷⁾, can be used to reduce the irradiation-induced swelling of metals and alloys for nuclear structural design considerations.

FIGURE UC-5. Observed Swelling of Irradiated Tungsten. Based on Immersion Density (O) and Dimensional (●) Measurements.

For the pure tungsten, TEM observations support the void-induced swelling.^(18,19) The swelling values reported here are considerably higher than previously reported⁽¹⁹⁾ due mostly to the higher fluence level.

From a broad viewpoint, the swelling data for tungsten appear similar to data presented for other metals.^(22,23) However, the swelling maximum occurs at an irradiation temperature of about $0.31 T_m$ instead of near the $0.44 T_m$ normally expected. The upturn in the swelling values at the highest temperature may be taken as an indication that a second swelling peak may occur with increasing irradiation temperature approaching the $0.44 T_m$ peak in agreement with previous results. The observed swelling maximum in these data coincides with the strong resistivity recovery peak occurring at the same temperature⁽²³⁾ where it is believed that free vacancies migrate.

A very important feature of the data, shown in Figure UC-4, is the difference in the swelling values obtained between the diameter measured of the swelling, $3\Delta d/d$, and the corresponding length measure, $3\Delta l/l$. The calculated swelling based on diametrical changes is about twice that determined from length measurements. However, because of the small change in diameter, the difference is barely significant at the 95% confidence level, although it does suggest possible anisotropic swelling behavior for the geometry of specimen used.

These data compare, for each irradiated temperature, the as-irradiated state with the state after the 2100°C anneal, instead of a comparison with the control specimen. The data are based on about 12 points on the recovery curve before recovery takes place and on 8 points on the plateau after recovery. Each point is the average of at least 3 individual micrometer readings. Error bands based on the statistics are shown in Figures UC-3 through UC-5. Similar differences between the diameter measure of swelling and other techniques have been⁽²³⁾, but not at a significant level.

Figure UC-5 shows the comparison between the measure of swelling based on immersion density measurement and the measure based on length and diameter ($2\Delta d/d + \Delta l/l$) changes. In this figure, the swelling is based on control specimens for both data sets. The difference at the highest irradiation temperature are quite large, but not significant at the 95 percent confidence level. Work is continuing on these materials and may explain the large variation.

IV. RADIATION EFFECTS ON NIOBIUM

A. DEFECT AGGLOMERATION IN NEUTRON-IRRADIATED NIOBIUM

V. Sikka and H. Jang

A study of defects in neutron irradiated Nb was carried out. A characterization of the microstructure of this material, irradiated in EBR-II at six different temperatures; 430, 580, 700, 800, 900 and 1050°C, is presented. Of particular interest will be to the observation of rafting at 430°C and voids at temperatures such as 900° and 1050°C.

Polycrystalline niobium of commercial purity was used for the present investigation. Specimens, 50 mils (1.27 mm) in diameter and 1.75 inches (45 mm) long, were irradiated in Row 7 of the EBR-II facility at six different temperatures; 430, 580, 700, 800, 900 and 1050°C to a fast neutron fluence of $\sim 1 \times 10^{22}$ n/cm² ($E > 1$ MeV). The fast neutron fluence in Row 7 of EBR-II was calculated from the neutron spectrum presented by J. L. Kamphouse, et al.⁽¹²⁾ The fast neutron flux densities for $E > 0.1$ MeV and $E > 1$ MeV were 2.3×10^{13} and 4.2×10^{12} n/cm² MW⁻¹ sec⁻¹, respectively. The displacement fluence and rate in terms of displacement per atom (dpa) and dpa sec⁻¹, based on the Kinchen and Pease model, were ~ 21 dpa and $\sim 1.2 \times 10^{-6}$ dpa sec⁻¹, respectively.

The electropolishing technique used to prepare thin foils suitable for TEM has been reported in another publication.⁽²⁴⁾ All electro-polished samples were observed using a JEM 200 kV transmission electron microscope equipped with a tilt-rotation stage and a specimen cold-finger.

The size and density of defect agglomerates (rafts and voids) were measured from high magnification (150,000X and 300,000X) micrographs, respectively. Void sizes were measured by using a Carl Zeiss particle size analyzer. The foil thicknesses were measured from extinction fringes.⁽²⁵⁾

The structure of unirradiated Nb is characterized by low dislocation density and the absence of any precipitates.

The specimens irradiated at 430°C contained all possible defect clusters, whereas those specimens irradiated at the higher temperatures contained predominantly voids. Typical micrographs for niobium irradiated at 430°C show defect agglomerates listed as follows: black spot clusters, similar to those reported by Loomis, et al.⁽²⁶⁾ for specimens irradiated at reaction ambient ($\sim 30^\circ\text{C}$) temperatures in the neutron fluence range of 3×10^{17} - 1.5×10^{19} n/cm²

($E > 0.1$ MeV) are seen. The black spot cluster size is ~ 50 - 60 Å and these represent only a small fraction of the total defect structure for specimens irradiated at $430 \pm 5^\circ\text{C}$. Molybdenum specimens neutron irradiated⁽²⁷⁾ at $430^\circ\text{C} \pm 5^\circ\text{C}$ to a fast neutron fluence of $\sim 1 \times 10^{22}$ n/cm² ($E > 1$ MeV), also showed similar black spot clusters.

Dislocation loops are observed; the average size of the loops is ~ 100 - 150 Å, similar to that observed during ambient temperature irradiations by Loomis, et al.⁽²⁶⁾ The loop density appears to be slightly greater than the black spot cluster density.

Defect cluster agglomerates, designated as rafts, are seen. These defect agglomerates are similar to those observed in neutron-irradiated molybdenum by Brimhall and Mastel⁽²⁾, Elen, et al.⁽²⁸⁾, and Sikka and Moteff⁽²⁷⁾; in tungsten, by Sikka and Moteff⁽²⁹⁾; and in vanadium by Elen, et al.⁽²⁸⁾ Rafts in niobium have not been reported before. The rafts lie on all four $\{111\}$ planes and appear to have $a/2\langle 111 \rangle$ Burgers vectors.

Rafts have been quantitatively characterized by their number density, and their average length and thickness. The raft number density was corrected to include those rafts which would be out of contrast. The raft thickness was measured from an edge-on orientation, which would represent the minimum thickness. The raft parameters of niobium, molybdenum and tungsten specimens irradiated simultaneously under identical conditions are summarized in Table UC-2.

TABLE UC-2
SUMMARY OF RAFT PARAMETERS FOR NEUTRON IRRADIATED Nb, Mo and W

Material	Irradiation ^(a)		Raft Parameters ^(b)			$r_g/A^{(c)}$	$r_c/A^{(d)}$
	Temperature °C	T/T_m	Density N_R , Raft cm ⁻³	Length l_R , Å	Thickness t_R , Å		
Nb	430	0.262	5.2×10^{13}	2200	290	8.0	2.8×10^{-3}
Mo	430	0.244	7.4×10^{14}	1600	100	4.9	3.3×10^{-3}
Mo-0.5Ti	430	0.244	1.5×10^{14}	1600	100	-	-
W	430	0.191	6.1×10^{14}	560	80	4.6	5.7×10^{-5}
W	580	0.232	3.0×10^{14}	1200	100	4.5	3.7×10^{-4}

(a) Specimens were irradiated in Row 7 of EBR-II reactor to a fast neutron fluence of $\sim 1 \times 10^{22}$ n cm⁻² ($E > 1$ MeV)

(b) Raft parameter data scatter is within 10 percent.

(c) $r_{\text{glide}} = A(\mu/\tau)^{1/4}$; where $A = [3bd^3/8(1-\nu)]^{1/4}$.

(d) $r_{\text{climb}} = A'(\mu e^{-E_D/kT}/T)^{1/5}$; where $A' = [(90/64)Zv_0d\delta^2b^2t/k]^{1/5}$.

Further types of microstructure observed at 430°C irradiation temperature include dislocation segments, which most likely have been produced by the coalescence and growth of small loops and/or includes the dislocation structure characteristics of the pre-irradiation condition, and the three-dimensional compact defect clusters designated as voids. These voids are in contrast to the planar defect clusters and the rafts which are three dimensional colonies of black spot clusters and/or loops.

Voids are observed at all irradiation temperatures. The void size, density and the void volume swelling data for this investigation and those reported in the literature are presently being evaluated. The void size, density and volume swelling observed in this investigation are plotted as a function of irradiation temperature in Figure UC-6. The void density and size remain essentially unchanged for irradiations in the temperature regime of 430°C to 700°C and show a decrease in density and an increase in size with an increase in temperature above 700°C.

FIGURE UC-6. Effect of Irradiation Temperature on Void Size, Density, and Volume Swelling of Niobium Irradiated to a Fast Neutron Fluence of 1×10^{22} n cm⁻² ($E < 1$ MeV).

The void size and density data for niobium neutron-irradiated in the EBR-II at six different temperatures are compared⁽²⁷⁾ in Figure UC-7 with those for molybdenum irradiated under exactly the same conditions. The void size and density remain constant for niobium in the temperature regime of 0.26 to 0.36 T_m , whereas for molybdenum, these parameters remain constant in the temperature regime of 0.24 to 0.34 T_m . The void density decreases and the size increases for irradiation temperatures greater than 0.36 T_m for niobium and 0.34 T_m for molybdenum.

FIGURE UC-7. Void Size and Density of Neutron Irradiated Niobium, Compared with Neutron Irradiated Molybdenum. (Specimens Simultaneously Irradiated in EBR II to a Fast Neutron Fluence of 1×10^{22} n cm⁻²).

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