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SEMI-ANNUAL PROGRESS REPORT
IRRADIATION EFFECTS ON REACTOR
STRUCTURAL MATERIALS
AUGUST, 1974 TO FEBRUARY, 1975

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
under Contract No. AT(45-1)-2170

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Compiled by T. T. Claudson

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SUMMARY

AEROJET NUCLEAR COMPANY

Results of strain controlled fatigue tests with tensile hold times at 700°C of irradiated (1.0 to 4.0×10^{22} n/cm², $E > 0.1$ MeV, 700-750°C) and unirradiated cold-worked Type 304 and 316 stainless steel are compared to annealed material. Although the number of irradiated cold-worked specimens was very limited, trends consistently show that the cold-worked stainless steel is damaged to a greater degree by neutron irradiation than annealed material at 700°C when tested in creep-fatigue. Type 316 shows greater damage than 304. Cold-work in unirradiated material shows little or no difference in creep-fatigue properties when compared to annealed stainless steel.

ATOMICS INTERNATIONAL

Proton irradiating 20% CW 316 at 600°C with a low flux to 1.5 dpa followed by a high-flux exposure to a total damage of 14 dpa resulted in less swelling than after a single high-flux exposure to 16 dpa, 2.4% compared to 7.5%.

Swelling of 25% CW 316 stainless steel after a fluence of 8×10^{22} n/cm² was absent at 500°C and was 3.3% at 600°C as determined by immersion density measurements.

Inconel 718, solution annealed and aged, was irradiated in EBR-II S/A X100 to a fluence of 4×10^{22} n/cm² ($E > 0.1$ MeV) at 500°C and 600°C. The swelling at 500°C was 0.1% and at 600°C no voids were formed.

GENERAL ELECTRIC COMPANY--FAST BREEDER REACTOR DEPARTMENT

Both examination of specimens and analysis of data were continued on Types 304 and 316 irradiated in EBR-II S/A X098A to 8×10^{22} n/cm², $E > 0.1$ MeV. The swelling rates for annealed material appear to be quite similar for both alloys if the comparison is made at the peak swelling temperatures. Transmission Electron Microscopy (TEM) examination of 20% Cold-Worked Type 316H shows that the dislocation substructure is measurably changed at all irradiation temperatures investigated (450°C to 675°C). Void formation and precipitation occurred at all temperatures. Detailed analysis of the Type 304 profilometry data shows that the steep axial gradients observed in the tubular Type 304 specimens were related more to axial fluence gradients than axial temperature variations. It can be expected that relatively large swelling rates of greater than 2.5% per 10^{22} n/cm², $E > 0.1$ MeV can occur in some austenitic stainless steels under certain combinations of irradiation conditions and/or preirradiation microstructure. These high swelling rates can be expected to commence at fluences of 5 to 10×10^{22} n/cm², after the void nucleation phase is complete.

NAVAL RESEARCH LABORATORY

The research program of the NRL Engineering Materials Division Thermostructural Materials Branch, involves a broad study of the effects of nuclear radiation upon materials. The program is sponsored by the Office of Naval Research, the U. S. Atomic Energy Commission, and the U. S. Army Engineer Power Group. Since research findings which apply to the objectives of one sponsoring agency are also of interest to the others, the overall program progress is reported herein. This report, covering research for the period, 31 August 1974-31 January 1975; includes: (1) the effect of hold time on fatigue crack propagation in neutron

irradiated 20% cold-worked Type 316 stainless steel; (2) high fluence assessments of 0.03% copper plate from 30-ton A533-B demonstration melt; (3) examination of microstructures of Inconel 718, Incoloy 800, PH13-8Mo, Mo and Nb after irradiation in EBR-II; (4) fluence dependence of ion damage in nickel; (5) ion-simulated irradiation-induced creep of reactor structural alloys; and (6) notch ductility and strength of Type 316 stainless steel submerged arc weld deposits.

I. ADVANCED REACTOR STRUCTURAL MATERIALS

A. Effect of Hold Time on Fatigue Crack Propagation in Neutron Irradiated 20% Cold-Worked Type 316 Stainless Steel

The effect of combined cyclic and static loading on crack propagation was investigated at 800°F (427°C) for 20% cold-worked Type 316 stainless steel. Pre- and postirradiation comparisons were made of the effect of 0, 0.1, and 1 minute hold times on crack growth rate as a function of stress intensity factor range. The results show that neither hold time nor neutron irradiation to a fast neutron fluence of 2.2×10^{22} n/cm² > 0.1 MeV produced a significant effect on crack growth behavior for the range of stress intensity factors investigated.

B. High Fluence Assessments of 0.03% Copper Plate From 30-Ton A533-B Demonstration Melt

The high fluence capabilities of improved composition A533-B Class 1 steel have been investigated with a series of five irradiation experiments at 530°F (277°C) to 600°F (316°C). The investigation used a 6-in. thick plate with a 0.03%Cu, 0.008%P, and 0.008%S impurity content. The plate was from the 30-ton commercial demonstration test melt originally made to verify improved radiation resistance with a reduced impurity content. Postirradiation Charpy-V and yield strength determinations indicated a strong material potential for service applications up to 2.5×10^{20} n/cm² > 1 MeV. Radiation embrittlement saturation was not observed.

II. RADIATION DAMAGE MECHANISMS

A. Examination of Microstructures of Inconel 718, Incoloy 880, PH13-8 Mo, Molybdenum and Niobium After Irradiation

Microstructures of four classes of materials, a precipitation hardening nickel base alloy, a solid solution hardened nickel base alloy, a martensitic alloy, and refractory metals were examined after irradiation to a fluence of 7.4×10^{21} n/cm² at 540°C (1004°C). Swelling was low in the alloys and the most significant observation was the coarsening of precipitates in the Inconel 718. Swelling in the refractories was about 0.4% even though the temperature was less than 0.3 of the absolute melting point.

B. Fluence Dependence of Ion Damage in Nickel

The effect of ion dose on void and dislocation structure was investigated for nickel irradiated at 575, 625, and 675°C. The damage levels studied were between 1.3 and 130 displacements per atom, and the damage dose rate was 7×10^{-2} dpa/sec. Comparison of the dose dependence of the swelling observed at 575 and 625°C, with the predictions of a theory for the rate of swelling due to void growth, indicated that the relationship between the swelling rates observed at these two temperatures could not be adequately explained by the model.

C. Ion-Simulated Irradiation-Induced Creep of Reactor Structural Alloys

An experimental program involving the ion-simulation of irradiation-induced creep is now under active development by NRL. Installation of the specially-designed apparatus and beam line components in Experimental Room No. 2 of the NRL Cyclotron is reported. Several experimental tests proved valuable in confirming some expectations and pointing towards necessary modifications.

An electronic control system has been designed and built to stabilize specimen temperature. The system is interfaced to an on-line Univac 6050 computer. Additionally, computer programs were developed and are now operational for recording six-parameter data, as well as performing on-line processing and displaying of measured or calculated variables.

III. ARMY REACTOR STUDIES

A. Notch Ductility and Strength of Type 316 Stainless Steel

Submerged Arc Weld Deposits

A series of four AISI Type 316 stainless steel weld deposits has been made by the submerged arc process for a follow-on study of pre- and postirradiation strength and Charpy-V notch ductility in support of the MH-1A reactor surveillance program. The welds represent differences in welding parameters and conditions and lot-to-lot differences in welding materials.

Significant differences in Charpy-V notch ductility were found among the weld deposits although tensile properties for the preirradiation condition were comparable. The choice of welding parameters was noted to have a significant effect on Charpy-V performance.

OAK RIDGE NATIONAL LABORATORY

Preliminary results of transmission electron microscopy examination of Type 316 stainless steel samples having variations in preirradiation microstructure are reported. These samples were irradiated to a maximum fluence of 7.8×10^{22} n/cm² (>0.1 MeV). When irradiated in the annealed condition to a fluence of 6.6×10^{22} n/cm² (>0.1 MeV), the density decrease was relatively small (0.59 and 1.53% at nominal irradiation temperatures of 500 and 600°C). In the sample irradiated at 600°C the void distribution was bimodal, and about 90% of the volume increase due to void formation was from large voids associated with massive precipitates. Void formation in the 20% cold-worked samples and in samples that were 20% cold-worked and aged at either 500 or 600°C for 10 hours prior to irradiation was inhomogeneous. In previous experiments samples having these same heat treatments showed no evidence of void formation at about 3×10^{22} n/cm² (>0.1 MeV). A tentative conclusion is that the preirradiation heat treatments do not significantly reduce the swelling resistance of the 20% cold-worked structure but may offer some advantages as concerns ductility and fracture behavior.

Results of diameter measurements on pressurized 20% cold-worked Type 316 stainless steel tubes irradiated at 550°C to 2.4×10^{22} n/cm² (>0.1 MeV) are reported. Deformation during irradiation was larger than expected from a simple addition of thermal creep strain (measured ex-reactor) and irradiation creep strain calculated from existing correlations. Preliminary examination and data analysis from swelling driven fracture samples indicate (1) that the technique can be used to measure the in-reactor fracture strain; (2) the in-reactor fracture strain of Type 304 stainless steel thimble material is larger than the postirradiation value;

however, the fracture mode remains intergranular; and (3) the in-reactor deformation rates are related to the swelling rate.

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 Type 316 stainless steel samples irradiated in HFIR to fluences approaching 10^{23} n/cm² has yielded results that are different in important respects from irradiations to equivalent fluences in fast reactors. Samples of the irradiated samples were successfully thinned remotely in the hot cells and TEM examination initiated. Preliminary examination showed cavities in all samples examined with the usual dependence (qualitatively) of cavity size and concentration on irradiation temperatures. There was also extensive precipitation in the irradiated samples, with marked association between cavities and precipitates. Dislocation loops were not found in any of the samples examined. Only line segments were present, with these pinned at precipitates and cavities. In general, we have found for irradiation temperatures near 550 and 680°C grain boundaries are not sites of enhanced cavity concentrations.

PACIFIC NORTHWEST LABORATORIES

The void microstructure has been analyzed in ion bombarded vanadium. Preinjected helium promotes void formation when the ion dose rate is low and in the presence of a high dislocation density. For a high dose rate in annealed material, helium was found to be not necessary for void nucleation. The results are in qualitative agreement with current nucleation theories.

UNIVERSITY OF CINCINNATI

Bulk density measurements performed on Mo, Mo-0.5Ti, Nb, Nb-1Zr, W and W-25 Re specimens irradiated at six temperatures in the range of 430 to 1050°C to a fast neutron fluence of 1×10^{22} n/cm², $E_n > 1$ MeV show that the alloying element influences the swelling behavior of the three base metals in three different ways.

Molybdenum specimens were irradiated with vanadium ions at dpa levels similar to that used in the neutron irradiated specimens. Swelling data based on TEM observations show good agreement with the bulk density measurements on the same material as reported above. The tendency to form a double swelling peak with respect to irradiation temperature for the ion irradiations is consistent with observations of the neutron irradiated molybdenum. The swelling based on TEM observations of the neutron irradiated specimens is considerably lower than the bulk density measurements.

It is proposed that the superlattice observed in molybdenum and tungsten be used as an in-reactor temperature monitor for capsule irradiations in a manner similar to the use of SiC as temperature sentinels.

HANFORD ENGINEERING DEVELOPMENT LABORATORY

I. IRRADIATION DAMAGE TO REACTOR MATERIALS

A. Low Temperature Creep of 20% Cold-Worked Type 316 Stainless Steel

Creep curves obtained from a single specimen of 20% cold-worked Type 316 stainless steel tested at 20,000 psi over a series of progressively higher temperatures (850, 1000, 1100°F) show that creep strain increases with increasing temperature. The introduction of only 0.06%

creep strain during the 850 and 1000°F loadings appears to significantly affect the creep response at 1100°F. A simple strain hardening rule does not fully account for the effect of prior strain history. Creep response after unloading exhibits measurable strain recovery at all temperatures and is consistent with an anelastic component in the total creep deformation. The magnitude of recovered strain is of the order of 0.02% at both 1000 and 1100°F. To a first approximation, creep response after reloading is consistent with the reintroduction of anelastic strain recovered during the prior unload and the continued accumulation of creep strain as if unloading had not occurred. Test results continue to demonstrate the applicability of the microwave extensometer for sensitive, precise creep strain measurements.

B. Effect of Irradiation on the Mechanical Properties of 20% C. W. Type 316 Stainless Steel Prototypic Cladding

Tensile, simulated transient and stress-rupture tests were conducted on 20% C. W. Type 316 stainless steel cladding material irradiated at 860 to 1050°F at fluences up to 3.6×10^{22} n/cm² (E>0.1 MeV). Data for the region of 900 to 1745°F were correlated with a Larson-Miller parameter. Test results show that increasing the neutron fluence produces decreases in loadbearing capability and ductility.

C. Test Temperature and Notch Effects on the Tensile Behavior of Highly Irradiated EBR-II Duct Thimble Material

The effects of test temperature, strain rate, fluence and notch geometry on the tensile properties of EBR-II irradiated, annealed Type 304 stainless steel have been investigated. In general, increasing test temperature over the range of room temperature to 1400°F is accompanied by a decrease in both strength and elongation independent of fluence level. Increasing strain rate from 2.67×10^{-3} /min to 2.67/min at the high fluence levels, $\sim 10^{23}$ n/cm², results in a slight increase in strength and a more pronounced increase in elongation. Strength increases and elongation decreases rapidly with fluence up to $\sim 3 \times 10^{22}$ n/cm² (E>0.1 MeV). Above this fluence level tensile parameters become relatively fluence independent. Notch sensitivity was exhibited only under conditions of intergranular fracture in material irradiated beyond $\sim 4 \times 10^{22}$ n/cm² (E>0.1 MeV). Intergranular fracture is the dominant mechanism above about 1100°F.

D. Thermal Creep in Pressurized Tubes of 20% Cold-Worked Type 316 Stainless Steel

Analytical equations describing thermal creep deformation in pressurized tubes of 20% cold-worked Type 316 stainless steel were formulated. These creep equations predict creep strain as a function of stress, time and temperature, and are experimentally verified from 825 to 1400°F for times to 2400 hours over the stress range from zero to as high as 60,000 psi.

E. Irradiation Creep in Cold-Worked Type 316 Stainless Steel

The results of three irradiation periods extending to a peak neutron fluence of 3×10^{22} n/cm² (E>0.1 MeV) has revealed the first evidence that in-reactor creep is temperature dependent. The strain data, after being normalized to stress and fluence or atom displacements, was found to increase with temperature in excess of that measured in thermal environments

before neutron irradiation. The stress dependence was found to continue in a linear fashion, but the strain increased nonlinearly with fluence. The fluence dependence of strain was described by a temperature dependent hyperbolic tangent function.

II. MATERIALS ANALYSIS AND APPLICATION

A. Full Power Dosimetry Experiment in the EBR-II

Six B-19 subassemblies located at 2D1, 4C3, 4E1, 7F4, 8F4 and 8F6 for the Full Power Dosimetry Experiment were irradiated for 8 full power days in the EBR-II during Run 75D for a total of 582 MWD. All the very short half-life monitors were counted by January 25, 1975. Completed radiometric results on the $^{63}\text{Cu}(n,\gamma)$ ^{64}Cu were made available for analysis on January 29, 1975.

B. Helium Production Cross-Sections

Helium productions for various materials were estimated using the recently issued ENDF/B-Version IV cross-sections and compared with measured data. Greatly improved agreement with measurement is noted for nickel which is the main contributor to helium production in stainless steel, but improved evaluations are still needed for other elements and isotopes.

C. LMFBR Structural Materials Evaluation

Tensile properties of neutron irradiated FTR component structural materials Inconel 718 (to 1×10^{22} n/cm², $E > 0.1$ MeV, at 760 and 1100°F), Inconel 600 (to 2.5×10^{21} n/cm², $E > 0.1$ MeV, at 700°F), and A-286 (to 2.5×10^{21} n/cm², $E > 0.1$ MeV, at 800°F) have been determined. The irradiated materials exhibited good strength and ductility. Irradiations of the Inconel 600 and 718 to higher fluence levels is continuing.

D. Subcritical Crack Growth

The effect of several elevated temperature operating environments (e.g. air, nitrogen, liquid sodium) and vacuum on the fatigue-crack growth behavior of reactor structural materials is being determined. Materials tested to date include annealed Type 304 stainless steel, annealed Inconel 600, and precipitation-heat-treated Inconel 718. Testing is continuing but the results to date indicate that at elevated temperature, air is the most aggressive environment, followed by nitrogen, and that vacuum and liquid sodium are the least aggressive (most inert).

III. LMFBR STRUCTURAL MATERIALS

A. FFTF/LMFBR Weld Materials Properties

Trend curves describing the temperature dependence of tensile properties have been formulated for several weld-deposited austenitic stainless steels and also for wrought Type 304. Ratios of elevated-temperature properties to room-temperature properties were fitted to polynomial expressions in temperature by regression analysis. Trend curves were established for 308/308L, CRE 308 and 16-8-2 as well as for all the weld metal data. The results showed that the degree of correlation between predicted and observed properties was dependent upon variations in weld process and parameters but that the ratio trend curve approach yielded a useful degree of correlation of the elevated-temperature properties even without considering all the complex features of the weld-deposited materials.

B. Spatial Dependence of Void Growth Rates in the Irradiation of Thin Foils--Theory and Calculations

Theoretical calculations have been made of the depth of surface influence on C_v and growth fluid (ξ) for typical HVEM irradiation conditions. The space dependence of void growth fluid is the true measure of the surface influence on void growth and for this quantity the surface influence extends to greater depths than for C_v . For low dislocation densities representative of preirradiation microstructures, the surface extends into the central foil region. As the dislocation density quickly increases due to irradiation, the surface influence is reduced to depths comparable to observed denuded zones.

C. Evaluation of 20% Cold-Worked 316 Swelling Correlation

Electron simulation experiments have shown that the steady-state swelling rates do not vary appreciably among the various heats of 20% CW 316, and any difference in swelling behavior must be due to variations in the bulk-integrated incubation fluence. Recently acquired data were analyzed to determine the range of incubation parameters $\tau(T)$ necessary to reconcile the new data set with current design predictions. Although $\tau(T)$ required modifications ranging from 10 to 40% above previous estimates, it was possible to define a range of $\tau(T)$ that covered the data field well. The impact of this variation on Demonstration Plant and FTR design predictions is small, however. Whereas the previous design correlation employed an uncertainty bond ranging from the nominal equation to 50% of the nominal at all fluences, the new correlation employs an uncertainty bond which approaches the nominal at high fluences.

D. Development of Solution-Annealed 316 Swelling Correlation

A swelling equation for solution-annealed 316 stainless steel was developed for use in the LIFE-III calibration activity for fuel pin modeling. A bilinear swelling law was employed. This correlation includes data from stressed fuel pin cladding. Comparison with recent fuel pin swelling data showed good agreement with predictions of the equation above 550°C. Below 550°C the new data suggest an overprediction of swelling.

E. Dose Concepts for Charged Particle Simulation Experiments

The calculational tools available for computation of ion displacement dose profiles have been reviewed and found to yield essentially consistent results, with only one exception which is still under study. These computer codes have been used to determine the depth-dependence of cascade mean size and the compositional variation of dose profiles. The effects of irradiation temperature on the modification of the effective dose concept are currently being studied. Void growth fluids at early stages of the irradiation are very sensitive to the influence of the front surface and to the material beyond the ion range. This influence becomes less important later in the irradiation, when the higher densities of dislocation and void sinks screen external influences.

F. Development of an Electron Beam Welding Process for the Reencapsulation of Radioactive Specimens

Specimens from the Metallurgical Variables experiment (subassembly X-089), previously irradiated to a peak fluence of 4×10^{22} n/cm², have been encapsulated into subcapsules for reirradiation to a goal peak fluence of 1×10^{23} n/cm² ($E > 0.1$ MeV). The welding procedure developed for this encapsulation utilizes an electron beam welder. The welds obtained from

this process are comparable in design and quality to those welds which could be obtained using nonirradiated specimens. Development of this procedure makes possible the continued application of reencapsulation techniques for EBR-II experiments.

G. A Relationship Between Equivalent Chromium Content and Irradiation-Induced Swelling in 316 Stainless Steel

A correlation is noted between equivalent chromium content and resistance to irradiation-induced swelling in various 316 stainless steel specimens which have slightly different chemical compositions. Several examples are cited where an increased concentration of an α -stabilizing minor constituent results in decreased swelling. It is shown that the relative swelling resistance of alloys having the same carbon and equivalent nickel contents is higher for those alloys with the higher equivalent chromium content.

H. Experimental Evidence for Stress Enhanced Swelling

Radiation induced void formation or swelling in reactor core structural materials has been recognized as being an important phenomenon and one which must be considered in the design of LMFBRs. Experimental and theoretical studies have been conducted to quantify the swelling behavior of metals and alloys. The most abundant source of specimens for swelling analyses has been reactor core components which have received high exposure to radiation and therefore display measurable levels of swelling.

Theoretical studies have predicted that the hydrostatic stress in a reactor core component may affect the swelling rate. Because the reactor components from which most high fluence swelling data have been obtained are not free of stresses, there remains the possibility that the observed swelling behavior reflects some effect of stress. Unfortunately, the stress histories of such reactor components from which swelling data have been extracted have not been sufficiently well known to allow a clear assessment of the effect of stress on swelling.

An experiment was therefore devised to obtain swelling data under conditions where the stress is known. Possible effects on swelling due to chemical and mechanical interactions between the structural material and fissile material, such as prevail in fuel pins, were avoided by irradiating internally pressurized tubing. Some initial results of such an experiment are described in this report.

Aerojet Nuclear Company

FATIGUE AND CREEP-FATIGUE OF IRRADIATED AND UNIRRADIATED TYPE 304 AND 316 STAINLESS STEEL

G. E. Korth and M. D. Harper

I. EFFECTS OF COLD WORK ON THE CREEP-FATIGUE BEHAVIOR OF IRRADIATED STAINLESS STEEL

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 annealed in a creep-fatigue test at 700°C. Only trends could be established since the number of irradiated specimens with cold-work was very limited.

Hourglass specimens were fabricated from 5/8 in. diameter Type 304 (HT 55697) and 316 (HT 65808) stainless steel. These materials were from AEC standard heat stockpiles and detailed material history⁽¹⁾ and composition⁽²⁾ have been previously documented. Irradiation pretreatment involved three conditions: (1) solution annealed, (2) cold-worked by swaging at room temperature to 11% reduction in area, and (3) CST, chilled-swaged-tempered (27% reduction in two steps) - LN₂ chill, swage 11% reduction in area, anneal 16 hours at 700°C, LN₂ chill, swage 16% additional reduction in area, anneal 16 hours at 760°C. Hardness values for the three conditions are shown in Table ANC-1.

TABLE ANC-1
HARDNESS VALUES OF SPECIMEN MATERIAL
AFTER THREE PREIRRADIATION TREATMENTS

	<u>Type 304</u>	<u>Type 316</u>
Annealed	HRB 77	HRB 84
11% CW	HRB 92	HRB 98
CST	HRB 88	HRB 98

Irradiated specimens were subjected to neutron fluences of $1.0 - 4.0 \times 10^{22}$ n/cm², E>0.1 MeV in EBR-II at 700-750°C. Thermal controls were aged at 750°C for 8760 hours to approximate time and temperature in the reactor. All tests were conducted in air at 700°C in strain controlled fully reversed push-pull axial tests. Other test and irradiation details can be found elsewhere.⁽²⁾

Previous fatigue tests^{(2),(3)} without hold times at lower temperatures and fluences have shown cold-worked and annealed material to compare quite closely in both the irradiated and unirradiated conditions. Current tests reported here, with the limited number of irradiated CW specimens, indicate that at 700°C and 10^{22} n/cm² fluence levels the introduction of hold times reduces the cyclic life below that of annealed material. The annealed Type 316 data was taken from previous work.⁽⁴⁾ Type 316 shows more of a reduction than does the Type 304 stainless steel.

FIGURE ANC.1. Fatigue Behavior with Tensile Hold Times of Irradiated and Unirradiated Type 304 Stainless Steel in the Annealed, 11% Cold-Work and Chilled-Swaged-Tempered (CST) Conditions.

Time-to-fail versus cycles-to-fail plots are shown in Figure ANC.1 for 304 stainless steel and in Figures ANC.2 and ANC.3 for 316 stainless steel. Tabulated data is given in Table ANC.2, where

$\Delta\epsilon_t$ = total strain range	N_0 = cycles at first sign of load decrease (crack initiation)
$\Delta\epsilon_p$ = plastic strain range	N_5 = cycles at 5% drop in load range
$\Delta\sigma$ = stress range, peak to peak, at $N_f/2$	N_f = cycles at failure (specimen broken in two)
σ_t = tensile amplitude, zero to peak, at $N_f/2$	
$\sigma_{t \text{ min}}$ = tensile amplitude at end of hold period at $N_f/2$	

TABLE ANC.2

STRAIN FATIGUE BEHAVIOR WITH TENSILE HOLD TIMES OF IRRADIATED AND
UNIRRADIATED TYPE 304 and 316 STAINLESS STEEL TESTED AT 700°C

Spec No.	Material & Condition		Fluence*	$\Delta\epsilon_t\%$	Properties at $N_f/2$						N_o	N_5	N_f	Time to Fail, h	Tensile Hold, h	Strain Rate, s ⁻¹	
					$\Delta\epsilon_p\%$	$\Delta\sigma$	σ_t		σ_t, min								
					ksi	MPa	ksi	MPa	ksi	MPa							
24-D	316	Annealed	Control ^a	2.40	2.00	85.5	589.5	41.7	287.5	----	----	----	----	153	2.55	0	8x10 ⁻⁴
63-D	316	Annealed	2.4	2.44	2.04	85.3	588.1	41.2	284.1	----	----	----	----	86	1.46	0	8x10 ⁻⁴
E-37	316	11% CW	Control ^b	2.43	2.00	95.2	656.4	46.8	322.7	----	----	176	195	249	4.32	0	8x10 ⁻⁴
30-D	316	Annealed	Control ^a	2.38	2.21	75.7	521.9	35.8	246.8	13.1	90.3	50	53	57	6.65	0.1	8x10 ⁻⁴
65-D	316	Annealed	2.3	2.29	2.05	75.4	519.9	36.2	249.6	13.7	94.5	16	18	21	2.44	0.1	8x10 ⁻⁴
D-93	316	Annealed	Control ^b	2.35	2.13	87.5	603.3	44.6	307.5	16.9	116.5	30	39	47	46.05	1.0	8x10 ⁻⁴
E-36	316	11% CW	Control ^b	2.38	2.16	91.5	630.9	46.7	322.0	19.3	133.1	40	48	52	52.18	1.0	8x10 ⁻⁴
E-31	316	11% CW	Control ^c	2.37	2.13	89.4	616.4	45.4	313.0	17.1	117.9	----	----	38	40.90	1.0	8x10 ⁻⁴
E-20	316	11% CW	1.96	2.35	2.09	90.1	621.2	42.3	291.7	13.5	93.1	----	----	3	3.03	1.0	8x10 ⁻⁴
D-92	316	Annealed	Control ^d	2.34	2.15	79.0	544.7	39.8	274.4	11.4	78.6	----	----	23 ^e	104.66	5.0	8x10 ⁻⁴
D-20	316	Annealed	1.1	2.35	2.14	82.9	571.6	41.8	288.2	12.8	88.3	11	11	12	59.45	5.0	8x10 ⁻³
B-41	304	11% CW	Control ^b	1.98	1.72	77.7	535.7	38.1	262.7	----	----	488	523	532	1.77	0	4x10 ⁻³
B-42	304	11% CW	Control ^b	2.00	1.82	70.2	484.0	34.9	240.6	20.4	140.7	262	277	302	31.01	0.1	4x10 ⁻³
B-16	304	11% CW	1.63	2.00	1.75	71.6	493.7	35.6	245.5	27.6	190.3	35	37	40	39.47	1.0	4x10 ⁻³
F-36	316	CST	Control ^b	1.02	0.68	92.4	637.1	44.7	308.2	----	----	1,561	1,635	1,745	2.60	0	4x10 ⁻⁴
23-D	316	Annealed	Control ^a	1.01	0.68	70.7	487.5	31.7	218.6	----	----	----	----	1,408	9.87	0	8x10 ⁻⁴
62-D	316	Annealed	1.8	1.05	0.72	71.0	489.5	34.9	240.6	----	----	----	----	526	3.83	0	8x10 ⁻³
B-44	304	11% CW	Control ^b	1.01	0.75	65.8	453.7	31.5	217.2	----	----	1,523	1,646	1,825	2.72	0	4x10 ⁻³
C-41	304	CST	Control ^b	1.00	0.71	64.3	443.3	32.8	226.1	----	----	1,370	1,415	1,429	2.12	0	4x10 ⁻³
D-91	316	Annealed	Control ^b	1.00	0.76	65.4	450.9	31.7	218.6	18.5	127.6	398	424	530	55.08	0.1	8x10 ⁻³
F-37	316	CST	Control ^b	1.00	0.71	96.2	663.3	45.2	311.6	25.8	177.9	277	288	289	28.60	0.1	4x10 ⁻³
F-16	316	CST	1.45	1.00	0.75	93.0	641.2	46.7	322.0	30.5	210.3	25	28	28	2.77	0.1	4x10 ⁻³
B-45	304	11% CW	Control ^b	1.00	0.79	61.7	425.4	30.2	208.2	12.1	83.4	837	856	874	87.83	0.1	4x10 ⁻³
C-45	304	CST	Control ^b	1.00	0.70	67.4	464.7	32.2	222.0	17.2	118.6	605	664	678	68.19	0.1	4x10 ⁻³
C-16	304	CST	1.04	1.00	0.79	64.0	441.3	31.8	219.3	15.4	106.2	156	168	183	18.10	0.1	4x10 ⁻³
A-44	304	Annealed	Control ^b	1.00	0.78	59.5	410.2	29.4	207.7	16.7	115.1	1,108	1,130	1,186	119.20	0.1	4x10 ⁻³
F-38	316	CST	Control ^b	1.04	0.76	85.2	587.4	40.6	279.9	15.1	104.1	93	119	134	131.41	1.0	4x10 ⁻³
A-20	304	Annealed	1.35	1.00	0.85	52.6	362.7	25.6	176.5	14.0	96.5	110	114	114	113.25	1.0	4x10 ⁻³
B-43	304	11% CW	Control ^b	1.01	0.86	56.3	388.2	27.4	188.9	12.7	87.6	253	318	319	314.58	1.0	4x10 ⁻³
B-20	304	11% CW	1.34	1.00	0.87	56.3	388.2	26.9	185.5	13.1	90.3	50	61	73	71.93	1.0	4x10 ⁻³
C-20	304	CST	1.10	1.01	0.84	60.3	415.8	29.4	202.7	9.4	64.8	95	98	99	97.57	1.0	4x10 ⁻³
C-43	304	CST	Control ^b	1.00	0.76	64.6	445.4	31.0	213.7	8.8	60.7	226	243	255	252.23	1.0	4x10 ⁻⁴
25-D	316	Annealed	Control ^a	0.54	0.26	59.7	411.6	27.8	191.7	----	----	----	----	4,066	15.25	0	8x10 ⁻⁴
70-D	316	Annealed	1.3	0.58	0.15	73.7	508.1	86.6	252.3	----	----	----	----	1,730	6.97	0	8x10 ⁻⁴
E-38	316	11% CW	Control ^b	0.60	0.30	68.6	473.0	32.9	226.8	----	----	3,489	3,594	3,654	14.40	0	8x10 ⁻⁴
E-39	316	11% CW	Control ^b	0.61	0.79	70.5	486.1	34.0	234.4	----	----	2,807	2,915	3,025	11.80	0	8x10 ⁻⁴
D-42	316	Annealed	Control ^b	0.59	0.39	52.6	362.7	24.9	171.7	18.6	128.2	2,000	2,350	2,500	35.20	0.01	8x10 ⁻⁴
69-D	316	Annealed	2.0	0.58	0.44	52.9	364.7	26.4	182.0	18.8	129.6	330	353	374	5.24	0.01	8x10 ⁻⁴
D-41	316	Annealed	Control ^b	0.57	0.41	49.5	341.3	22.2	153.1	9.4	64.8	484	492	518	53.80	0.1	8x10 ⁻⁴
D-90	316	Annealed	Control ^d	0.60	0.42	55.4	382.0	27.6	190.3	14.7	101.4	----	----	262 ^e	257.10	1.0	8x10 ⁻⁴
D-16	316	Annealed	1.5	0.59	0.40	50.9	350.9	25.3	174.4	12.1	83.4	----	----	116	116.80	1.0	8x10 ⁻⁴
68-D	316	Annealed	3.7	0.58	0.45	47.8	329.6	23.1	159.3	13.9	95.8	63	67	71	71.28	1.0	8x10 ⁻⁴
E-16	316	11% CW	1.93	0.58	----	----	----	----	----	----	----	----	----	20	20.27	1.0	8x10 ⁻⁴
A-41	304	Annealed	Control ^b	0.56	0.43	40.1	276.5	20.8	143.4	11.6	80.0	----	----	601 ^e	594.40	1.0	8x10 ⁻⁴
A-16	304	Annealed	1.58	0.58	0.43	42.0	289.6	20.2	139.3	9.2	63.4	144	163	189	187.26	1.0	8x10 ⁻⁴

* Fluence given in units of 10²² n/cm², E>0.1 MeV, Irradiated at 700-750°C, unirradiated control specimens aged as noted.

^a 500°C for 1541 hrs

^c 400°C for 8760 hrs

^e Test terminated before failure

^b 500°C for 8750 hrs

^d 600°C for 3096 hrs

FIGURE ANC.2. Fatigue Behavior with Tensile Hold Times for Irradiated and Unirradiated Type 316 Stainless Steel Tested at 700°C at a Trial Strain Range of 2.4% (a) and 0.50% (b). (Irradiated to a fluence of 1.0 to $1.5 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$ at 700-750°C).

FIGURE ANC.3. Fatigue Behavior with Tensile Hold Times for Irradiated and Unirradiated Type 316 Stainless Steel Tested at 700°C. (Irradiated to fluence of 1.0 to $1.5 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$ at 700-750°C).

The general curve trends were established with unirradiated and annealed data. The irradiated-cold-work effect can then be seen by the deviation from the trends of the very limited irradiated CW data when compared to annealed material. The data show there is little difference between the creep-fatigue behavior of cold-worked and annealed material in the unirradiated condition, but in the irradiated condition the cold-worked stainless is inferior to the annealed material, especially the Type 316 stainless steel. In the case of Type 304, the irradiated CST stainless indicated a slight superiority to the 11% CW material even though the unirradiated CST material showed just the opposite. Direct comparisons with CST and 11% CW were not available with the Type 316 stainless. In Figure ANC.2b two Type 304 stainless data points are plotted with the Type 316 data to illustrate the higher creep-fatigue resistance of Type 304. This same result has also been observed at 593°C. ⁽⁵⁾

Although the data is very limited, trends consistently show that cold-worked stainless steel is damaged to a greater degree by neutron irradiation than annealed material at 700°C when tested in creep-fatigue. The observation that the cold-worked Type 316 stainless is damaged more than Type 304, and considering the temperatures involved, leads one to the

conclusion that sigma phase may play a significant role in the increased damage. Transmission electron microscopy would probably verify or discount this hypothesis, but has not been performed.

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Atomics International

I. THE EFFECT OF DAMAGE RATE ON SWELLING OF 20% CW TYPE 316 STAINLESS STEEL IRRADIATED WITH PROTONS

D. W. Keefer, A. G. Pard, K. R. Garr, M. M. Nakata

A specimen was irradiated at 600°C with protons in three sections as shown in Table AI.1. All three sections received an initial low-flux exposure and two of the sections received an additional high-flux exposure. The results are also shown in Table AI.1.

TABLE AI.1
THE EFFECT OF DAMAGE RATE ON VOID FORMATION AT 600°C

Sample	Section	Helium (appm)	Damage Rate (dpa/s)	Damage (dpa)	Total Damage (dpa)	Swelling [$\Delta V/V_0$, %]	Void Density (/cm ³)	Ave. Size (nm)	Min. Size (nm)	Max. Size (nm)
310-20	1	8.9	5.0×10^{-5}	1.4	1.4	None	None			
310-20	2	8.9	4.6×10^{-5}	1.5	6.8	0.16	5.6×10^{14}	16	3.8	34
			4.1×10^{-4}	5.3						
310-20	3	8.9	5.0×10^{-5}	1.4	14	2.4	1.5×10^{15}	28	8.2	72
			4.7×10^{-4}	13						
240-20		6.1	2.5×10^{-4}	16	16	7.5	1.6×10^{15}	40	10	75

For a comparison, void data from another specimen (240-20) which was exposed to only a high flux of protons at 600°C is included. Proton fluxes and fluences are given in Table AI.2.

TABLE AI.2
PROTON FLUX AND FLUENCE AT 0.75 MeV

Sample	Section	Proton Flux (p/cm ² -s)	Proton Fluence (p/cm ²)	Total Proton Fluence (p/cm ²)
310-20	1	1.8×10^{14}	5.4×10^{18}	5.4×10^{18}
310-20	2	1.8×10^{14}	5.4×10^{18}	2.7×10^{19}
		1.6×10^{15}	2.2×10^{19}	
310-20	3	1.8×10^{14}	5.4×10^{18}	5.3×10^{19}
		1.7×10^{15}	4.8×10^{19}	
240-20*	-	1.6×10^{15}	1.1×10^{20}	1.1×10^{20}

Irradiating to 1.5 dpa at a low flux decreased the swelling observed after irradiation to a total of 14 dpa. (Compare 310-20, Section 3, with 240-20 in Table AI.1.) The decrease in swelling is due almost entirely to a smaller void size.

Nucleation of voids apparently continued in 310-20 at the high flux between 6.8 and 14 dpa. This implies the presence of mobile helium in the lattice during this part of the irradiation. Note that the nucleation may have ceased before the end of the irradiation since the minimum void size for Section 3 of sample 310-20 exceeds that of Section 2.

The swelling of Section 2 of sample 310-20 is also much less than the 1 to 2% that would be expected after a high-flux irradiation to the same dpa based upon our previous results. The lower swelling is due to reduced void size and density (see Annual Progress Report, AI-AEC-13131, August 30, 1974).

Section 2 was held ~5 hours at 600°C while Section 3 was being irradiated. This does not appear to have affected the void population. The size distribution is reasonably symmetric, and the minimum void size is small. However, this point must be checked in future experiments to validate the previous observations.

Figures AI.1 through AI.5 illustrate the void formation in Sample 310-20. The cold-worked structure causes the void formation to be inhomogeneous. Regions of high dislocation density contain fewer voids.

II. SWELLING OF 25% CW TYPE 316 STAINLESS STEEL

EBR-II S/A X100

K. R. Garr

Immersion density measurements were made by ANL-W on four cylindrical specimens which had received a fluence of 8×10^{22} n/cm² (E>0.1 MeV) along with eleven control specimens. The density changes are shown in Table AI.3. The average value of the densities of the control specimens was used to calculate the density changes of the irradiated specimens. The uncertainty of one standard deviation assigned to the density changes is due mainly to the fact that the density measurements were not made on the same specimen before and after irradiation.

TABLE AI.3

SWELLING IN 25% CW 316 STAINLESS STEEL AFTER
A FLUENCE OF 8×10^{22} n/cm² (E>0.1 MeV)

Capsule No.	Initial Temp. (°C)	Final Temp. (°C)	Density Change (%)
3	500	500	+0.1 ± 0.2
5	500	544	+0.0 ± 0.2
4	600	535	-1.0 ± 0.2
6	600	600	-3.3 ± 0.2

III. TEM OF INCONEL 718 FROM EBR-II S/A X100

A. G. Pard and K. R. Garr

Foil samples of Inconel 718 were irradiated in EBR-II S/A X100 to a fluence of 4×10^{22} n/cm² (E>0.1 MeV) at 500°C and 600°C. Prior to the irradiation, the samples were treated for 2 hours at (2h) 1000°C + 7h 720°C and furnace cooled to 620°C and held for 5h. Figures AI.6 and AI.7 illustrate the preirradiation microstructure. The precipitates are mostly γ'' with some γ' . The large particles in Figure AI.7 are carbides.

FIGURE AI.1. Sample 310-20 Section 1 (no voids were formed.)

AI-4

FIGURE AI.2. Void Formation in Sample 310-20, Section 2.

FIGURE AI.3. Void Formation in Sample 310-20, Section 2.

FIGURE AI.4. Void Formation in Sample 310-20, Section 3.

FIGURE AI.5. Void Formation in Sample 310-20, Section 3.

FIGURE AI.6. Inconel 718, Solution Annealed and Double Aged (2h 1000°C + 7h 720°C FC to 620°C, 5h).

FIGURE AI.7. Inconel 718, Solution Annealed and Double Aged (2h 1000°C + 7h 720°C FC to 620°C, 5h).

Figures AI.8 and AI.9 show the microstructure after irradiation at 500°C and 600°C. Voids were formed at 500°C but none were formed at 600°C. Faulted loops were present at both temperatures at a density of 10^{14} - $10^{15}/\text{cm}^3$. Counting and sizing of voids at 500°C gave the following results:

$\Delta V/V_0 = 0.1\%$, void density = $8 \times 10^{13}/\text{cm}^3$, and average void size = 26 nm. Voids were not observed any closer than about 700 nm to grain boundaries.

There was a precipitate and defect-free region, about 50 nm wide, along grain boundaries in the 600°C sample. In addition, there were larger clear areas originating at grain boundaries and the example seen in Figure AI.9 is not an isolated one. More carbide precipitation was observed than in the preirradiation condition.

FIGURE AI.8. Inconel 718 Irradiated to a Fluence of $4 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$).

FIGURE AI.9. Inconel 718 Irradiated to a Fluence of $4 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$).

General Electric Company

STRUCTURAL MATERIALS IRRADIATIONS

H. J. Busboom

The series of structural materials irradiation tests conducted by GE will provide design basis 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 USAEC (ERDA) 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 prototypical fuel cladding tubing at various temperatures and monitoring the growth of these tubes as a function of fluence. Subassembly X098A, which contains 20% cold-worked Type 316 stainless steel at $\sim 8 \times 10^{22}$ n/cm², E>0.1 MeV, was discharged from EBR-II following Run 67. The interim examination has been completed at ANL's hot fuel examination facility (HFEF) in Idaho, and selected materials have been reencapsulated for continued irradiation starting with Run 71. The irradiation history and results from the profilometry examination were presented in the last report in this series.⁽¹⁾

Transmission Electron Microscopy (TEM) disks were extracted from the 20% cold-worked Type 316H irradiated at the higher fluences. The results from these examinations along with micro immersion density results taken on similar TEM disks at HEDL (Courtesy of Howard Brager, HEDL) are shown in Table GE.1. The volume swelling as measured by immersion density and TEM are in close agreement with that determined by diameter measurements. Typical photomicrographs are shown in Figure GE.1.

TABLE GE.1
SUMMARY OF SWELLING RESULTS FOR 20% COLD-WORKED TYPE 316

SAMPLE IDENTITY	AXIAL LOCATION ⁽²⁾	TEMP OF IRRAD. (°C)	FLUENCE x 10 ²² E>0.1 MeV	DIA. MEAS.	MICRO-IMMERSION DENSITY	TEM ⁽¹⁾	VOID NUMBER DENSITY	MEAN VOID DIAMETER ⁽³⁾
LP2C-1	5.6	454	8.2	-0.48	-	0.04	8.5×10^{13}	190
LP2C-ID2	5.6	454	8.2	-0.48	0.13			
LP2C-2D4	4.0	454	8.0	-0.48	0.05			
LP2G-1	5.5	535	8.5	0.48	-	0.04	8.1×10^{13}	500
LP2G-ID2	5.6	535	8.5	0.48	0.42			
LP2G-2D4	4.0	531	8.2	0.48	0.26			
LP2K-1	5.5	609	8.5	2.00	-	2.6	5×10^{12}	1900
LP2K-ID2	5.6	609	8.5	2.00	2.30			1500
LP2K-2D4	4.0	601	8.2	1.40	1.60			
LP2R-1	5.5	689	8.9	-0.12	-	0	3×10^{13}	90
LP2R-ID2	5.6	689	8.9	-0.12	0.01			
LP2R-2D4	4.0	682	8.5	-0.12	0.10			

(1) Uncertainty in TEM $\pm$ 30%.

(2) Location

(3) Angstroms

BEFORE

450°C

525°C

600°C

675°C

FIGURE GE.1, 20% CW Type 316, Before and After $8 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$.

Voids were observed at all four temperatures. Mean void diameters were estimated for a series of micrographs taken from each specimen and were found to increase from $190\text{\AA}$ to $1900\text{\AA}$ with increasing temperature to 600°C . The 600°C specimen contained a bimodal distribution of void sizes: large voids, $\sim 2000\text{\AA}$, associated with intragranular dispersed carbide particles, and small voids, $\sim 160\text{\AA}$, distributed between the particles. The larger voids contributed most to the observed volume swelling. The 675°C specimen contained a small number of voids with a mean diameter of $\sim 90\text{\AA}$.

Significant numbers of precipitate particles were apparent at all temperatures, many of which were associated with voids. To the contrary, no precipitates were detected in the archive specimen cut from as-received tubing.

The dislocation substructure was noticeably changed at all temperatures. The archive specimen exhibited cellular dislocation arrays with dense tangles at cell walls typical of cold-worked austenitic stainless steel. These cellular features and tangles were not present in the specimen irradiated at 450°C , indicating that some recovery had occurred. Increasing the temperature of irradiation resulted in a marked decrease in dislocation density, with the remaining dislocations forming arrays indicative of polygonization. At 675°C , a well polygonized microstructure was evident.

A. DISCUSSION OF RESULTS FROM S/A X098A

In order to compare the swelling behavior between alloys, care must be taken to make this comparison at the peak swelling temperature for each alloy. To illustrate this point, the swelling for the solution annealed Types 304 and 316 were plotted versus fluence in Figure GE.2. For simplicity, straight lines were drawn between comparison data points. At 450°C ,

FIGURE GE.2. Swelling Rates for Annealed Type 304 and Type 316 in EBR-II.

it is apparent that Type 304 swells much more than Type 316, while at 600°C the reverse is true. Since the maximum swelling measured in this test (6.2%) was observed in Type 304, we can say that it swells more than Type 316 for the fluences investigated. This is in general agreement with other modest fluence neutron⁽²⁾ and ion⁽³⁾ irradiation experiments where such direct comparison between these alloys can be made. However, it is apparent from Figure GE.2 that at fluences in excess of 8×10^{22} n/cm², the difference in peak swelling for these alloys is not as great as first suggested if the comparison is indeed made at the peak swelling temperatures and at the higher fluences. Figure GE-2 also supports the generally accepted premise that the major difference between these alloys, in their swelling behavior at 450°C, results from a longer threshold fluence before measurable swelling occurs in Type 316⁽⁴⁾.

The major difference between the Types 304 and 316 irradiated in this experiment are in composition and solution annealing temperature. The composition differences include higher nickel and higher minor alloying element contents (mainly molybdenum) for the Type 316. Also, the chromium content is lower for the Type 316. Recent nickel ion bombardment simulation experiments have shown that these composition differences are all in the right direction to reduce swelling. The solution annealing temperature was higher for the Type 316 which has also been shown to reduce swelling in nickel ion bombardment⁽⁵⁾. This effect was thought to be associated with the amount of interstitial elements, principally carbon, in solid solution. The above explanation could also account for the slight swelling observed at the lower fluence ($\sim 3 \times 10^{22}$ n/cm², E>0.1 MeV) in the Garofalo heat-treated Type 316 material as compared with zero swelling in the solution annealed Type 316⁽¹⁾. The Garofalo heat treatment is known to precipitate carbides⁽⁶⁾ and thus reduce the amount of carbon left in solid solution. Recent results from France⁽⁷⁾ show that carbon in solution plays a rather important role in limiting alloy swelling in Type 316. The more carbon put into solution before irradiation, the lower the observed swelling.

A similar swelling response has been observed in an earlier GE fuel experiment where Type 304 cladding was irradiated in both the cold-worked and cold-worked plus heat-treated condition⁽⁸⁾. When both Type 304 materials were irradiated at similar fluence and temperature, the cold-worked plus heat-treated cladding exhibited much larger volume swelling than did the plain cold-worked cladding. ORNL workers⁽⁹⁾ have also observed modest swelling to 1% $\Delta V/V_0$ in Type 316 which was cold-worked and then recovered prior to irradiation. Similar material aged and then cold-worked exhibited density increases after irradiation.

The temperature at which peak swelling occurred for the Type 316 in S/A X098A was $\sim 600^\circ\text{C}$ and is similar to that observed by U.K. investigators⁽¹⁰⁾ for cold-worked M316 fuel cladding irradiated to fluence in excess of 5×10^{22} n/cm² in DFR. This similarity is shown in Figure GE.3 along with the only other available high neutron fluence swelling results which were generated in France.⁽⁷⁾ These latter results show the peak swelling temperature to be much lower (around 525°C) but they also exhibit a steep slope with increasing temperature above the peak swelling temperature. It should be noted that for solution annealed Type 316, the French see a second peak at $\sim 600^\circ\text{C}$. This may also exist in their cold-worked Type 316 but their present data are obtained only at lower temperatures. Difficulty in measuring in-reactor temperature is well recognized and some of the differences between data sets may arise from temperature uncertainties.

FIGURE GE.3. Swelling of Cold-Worked Type 316.

The TEM examination of the 20% cold-worked Type 316 presented earlier can be compared to similar examination of M316 fuel claddings from the U.K.⁽¹⁰⁾ and Type 316 fuel claddings from earlier GE F2 fuel experiments⁽¹¹⁾. Of particular interest is the similarity in the temperature dependence of void size as shown in Figure GE.4. Although the fluence varies from 4.3 to $8.5 \times 10^{22} \text{ n/cm}^2$, the trend of increasing void size with increasing temperature

FIGURE GE.4. Mean Void Diameters in Type 316 Fuel Cladding.

is similar for the three independent sets of data. The main point to be made here is that the temperature of irradiation calculated for the L-16 specimens agrees rather well with the cladding temperatures calculated from fuel rod experiments if one assumes void size to be mainly temperature dependent. Void number density, however, is not as well behaved due to the heterogeneity of the voids observed in the cold-worked materials. This was particularly true for the very low swelling specimens in the L-16 experiment, Table GE.1.

B. SWELLING OF TYPE 304

Measurements of metal swelling in the nonfueled Type 304 cladding tubes irradiated in S/A X098A have been analyzed in greater detail. The specimens were 2-3/8 inches long and cut from 1/4 inch diameter x 0.015 inch wall Type 304 tubing which was well characterized earlier⁽¹²⁾. Both annealed and cold-worked specimens were welded together to form a specimen train which fits concentrically into a Mark A-19A capsule tube. Each train was irradiated in Row 4 of EBR-II at one of four constant temperatures: 450, 525, 600, and 675°C. Diameter measurements were taken before and after fluences of 3.2 and 7.8×10^{22} n/cm², E>0.1 MeV. Details of the irradiation test have been described earlier⁽¹³⁾.

Typical postirradiation diameter profiles are shown in Figures GE.5 and GE.6. These

FIGURE GE.5. L-16 Profilometer Trace.

FIGURE GE.6. L-16 Profilometer Trace.

profiles are for specimens irradiated at the higher fluences at 450°C and 525°C. At first glance these profiles suggest that cold-work reduces the swelling of Type 304 at 450°C and can either increase or decrease the swelling at 525°C depending on the amount of cold-work. However, an important feature of these profiles is the swelling gradient along the individual specimens, and it was not readily apparent what caused them.

An evaluation of these gradients requires the flux and temperature profiles which are shown in Figure GE.7. The normalized flux profile was taken from the EBR-II Guide⁽¹⁴⁾. The

FIGURE GE.7. Temperature and Flux Profiles.

temperature profiles were calculated using the TIGER V code for two dimensional heat transfer. Attention is drawn to the local hot spots at the spacer joints between the different specimen materials.

Three points were taken for analysis from each specimen, the midpoint and one on each end just before the local hot spot. Fluence, temperature and volume swelling are tabulated in Table GE.2 for each point. Volume swelling was determined using the relation $\Delta V/V = 3\Delta D/D$. Both axial and radial in-pile positions were considered in the fluence calculations which also took into account the lower power than the 62.5 MW commonly quoted for EBR-II⁽¹⁵⁾.

The first step taken was to neglect the fluence variations and assume that the temperature gradient was the major contributor to the swelling gradient along each specimen. This temperature dependence for swelling is shown in Figure GE.8. Straight lines connect the midpoints for specimens from each condition: 0%, 10%, 20% and 30% cold-worked. The data scatter is considerable and in most cases the slope for points from each specimen differ from that determined from the midpoint points for specimens for each condition. Thus, it appears that the swelling gradients do not correlate simply with the temperature gradient along each specimen and it might be assumed that temperature did not contribute greatly to the variation in swelling along each specimen.

The second step taken was to neglect the temperature variation along each specimen and assume that the axial fluence gradient was the major contributor to the swelling gradient. This fluence dependence is shown in Figures GE.9 and GE.10. Straight lines can be drawn through the data for each condition which suggests that fluence was a major contributor to the observed swelling behavior.

The projected swellings at 450°C and 525°C can be extended to higher fluences based on the observed linear relations shown in Figures GE.9 and GE.10. Available high fluence

TABLE GE.2
LOCAL SWELLING IN TYPE 304 SPECIMENS

<u>MATERIAL</u>	<u>RADIAL ⁽¹⁾ POSITION</u>	<u>AXIAL ⁽²⁾ POSITION</u>	<u>TEMP.</u>	<u>FLUENCE ⁽³⁾</u>	<u>$\Delta V/V_0$</u>
	(in)	(in)	(°C)	(n/cm ²)	(%)
Annealed	7.10	9.0	451	3.11	1.62
	7.10	9.7	449	2.98	1.56
	7.10	10.3	446	2.85	1.50
	7.10	9.0	451	7.62	6.55
	7.10	9.7	449	7.30	6.15
	7.10	10.3	446	7.00	5.80
	6.52	9.0	527	3.21	0.72
	6.52	9.7	523	3.08	0.60
	6.52	10.3	520	2.95	0.48
	6.46	9.0	527	7.80	3.95
	6.46	9.7	523	7.47	3.40
	6.46	10.3	520	7.16	2.15
	6.13	9.7	590	3.14	0
	5.88	9.0	594	8.02	1.88
	5.88	9.7	589	7.68	1.77
	5.88	10.3	584	7.36	1.20
	5.53	9.0	666	3.23	0
	5.88	9.0	666	7.68	0
10% CW	6.13	7.3	606	3.50	0
	7.10	6.5	454	8.23	4.30
	7.10	7.3	454	8.13	4.20
	7.10	7.9	453	8.00	4.08
	6.46	6.5	535	8.42	5.90
	6.46	7.3	534	8.32	5.10
	6.46	7.9	532	8.19	3.80
	5.88	7.3	605	8.51	0
10% CW	5.53	7.3	685	8.78	0
20% CW	7.10	7.3	454	3.32	0
	6.52	7.3	534	3.43	0
	6.13	4.8	606	3.50	0
	5.53	7.3	688	3.59	0
	7.10	4.1	453	8.00	2.63
	7.10	4.8	454	8.15	2.82
	7.10	5.5	454	8.23	2.97
	6.46	4.1	532	8.19	2.55
	6.46	4.8	534	8.34	4.30
	6.46	5.5	535	8.42	5.10
	5.88	4.8	605	8.51	0
	5.53	4.8	685	8.72	0
30% CW	6.13	2.4	590	3.16	0
	7.10	1.7	446	7.00	1.29
	7.10	2.4	449	7.34	1.53
	7.10	3.0	451	7.61	1.90
	6.46	1.7	520	7.16	0.2
	6.46	2.4	524	7.51	1.1
	6.46	3.0	527	7.78	1.2
	5.88	2.4	590	7.81	0
	5.53	2.4	670	8.06	0

-
- (1) Distance from core center line.
(2) Distance from bottom of core.
(3) Fluence for Neutrons with Energies >0.1 MeV.

swellings from nickel ion-bombardment experiments⁽³⁾ have also been included in Figure GE.10 to illustrate the similarity in slope for both sets of data. It is commonly thought that the peak swelling rates in nickel-ion bombardment are similar to those from neutron bombardment although the peak swelling temperature is different⁽³⁾. These EBR-II results support this contention.

The major observation from this work is that the steady state fluence dependence of swelling in Type 304 is very temperature sensitive as shown in Table GE.3. Also, the swelling rate is sensitive to preirradiation microstructure at 525°C but not at 450°C. Similar high swelling rates have been observed earlier in Types 316 and 347 specimens which were exposed to sodium prior to neutron irradiation at ~500°C⁽¹⁾. Thus, it can be expected that relatively large swelling rates of greater than 2.5% per 10^{22} n/cm², E>0.1 MeV can occur in some austenitic stainless steels under certain combinations of irradiation conditions and/or preirradiation microstructure. These high swelling rates can be expected to commence at fluences of 5 to 10×10^{22} n/cm², after the void nucleation phase is complete.

TABLE GE.3
STEADY STATE SWELLING RATES IN TYPE 304

CONDITION	SWELLING RATE (% 10^{22} n/cm ²)		
	450C	525C	600C
Annealed	1.1	2.5	1.7
10% CW	1.0	10	-
20% CW	1.1	10	-
30% CW	1.1	-	-

FIGURE GE.8. Temperature Dependence of Swelling in Type 304 at $\sim 8 \times 10^{22} \text{ n/cm}^2$.

FIGURE GE.9. Fluence Dependence of Swelling in Type 304 Stainless Steel at 450°C.

FIGURE GE.10. Fluence Dependence of Swelling in Type 304 at 525°C.

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Naval Research Laboratory

I. ADVANCED REACTOR STRUCTURAL MATERIALS

A. EFFECT OF HOLD TIME ON FATIGUE CRACK PROPAGATION IN NEUTRON IRRADIATED 20% COLD-WORKED TYPE 316 STAINLESS STEEL

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The service conditions which austenitic stainless steel (SS) structural components of advanced nuclear systems are expected to experience include the combined effects of cyclic and static loading during neutron irradiation at sustained elevated temperatures. These conditions necessitate the consideration of the effects of the creep-fatigue interaction, thermal aging, and neutron irradiation on the elevated temperature fatigue crack growth behavior of the candidate materials.

Previous reports showed that fast neutron irradiation of annealed Types 304 and 316 SS produced an increase in fatigue crack growth rates during postirradiation tests at 800 and 1100°F (427 and 593°C) for low values of the stress intensity factor (ΔK).⁽¹⁾ Fatigue crack growth rates in irradiated 25% cold-worked Type 304 SS tested at 1100°F (593°C) were increased by fast neutron irradiation with a negligible effect of irradiation observed during 800°F (427°C) tests.⁽²⁾ Other NRL studies have shown that thermal aging at 1100°F (593°C) produced a slight decrease in fatigue crack growth rates of unirradiated annealed Types 304 and 316 SS while increasing the crack growth rates of 25% cold-worked Type 304 and 20% cold-worked Type 316 material at 1100°F (593°C).⁽³⁾

The effect of static tension loading (hold time) imposed on cyclic loading has been shown to produce factors of ten increase in crack growth rate of unirradiated 20% cold-worked Type 316 SS at 1100°F (593°C) for hold times of 0.1 minute and 1 minute.⁽⁴⁾ A recent NRL study showed that in thermally aged material, the increase in crack growth rate produced by a 1 minute hold time was found to be less than that produced by a 0.1 minute hold time in unaged material. Similar results have been obtained for thermally aged, annealed and 25% cold-worked Type 304 SS during hold time tests.⁽⁶⁾ However, few experiments have considered the effects of the creep fatigue interaction on crack propagation at elevated temperatures in neutron irradiated structural materials. The present report represents initial information from a study of this effect in fast neutron irradiated austenitic SS and nickel-base alloys.

The chemical composition of the Type 316 SS, the specimen design, and the general test procedures have been previously reported in detail.^{(1),(7)} Briefly, single-edge-notched cantilever specimens were cycled under zero-to-tension loading to a constant maximum level. Two loading wave forms were employed for the experiments: a sawtooth wave at a frequency of 10 cpm for the zero hold time tests and a truncated sawtooth wave form for the hold time tests. Static tension loads were imposed on the test specimens by holding the constant maximum load for times of 0.1 and 1.0 minutes. Induction heating was used to achieve and maintain the test temperature. All tests were conducted in laboratory air.

The neutron irradiated specimens used in this study were irradiated in flowing sodium in EBR-II subassembly X188, location 4F2, to a fluence of 2.2×10^{22} n/cm² >0.1 MeV. The subassembly midplane irradiation temperature was approximately 800°F (427°C). The unirradiated specimens tested for comparison with the irradiated specimens were not thermally aged; however, a set of controls is now being aged to approximate the reactor thermal exposure conditions. The experimental data were analyzed to yield crack growth rate, da/dN, as a function of the stress intensity factor range, ΔK .

Figure NRL.1 summarizes the results for the effect of hold time on fatigue crack growth in neutron irradiated 20% cold-worked Type 316 SS tested at 800°F (427°C). The data show that, for the irradiation and test conditions employed in this work, the effect of 0.1 and 1 minute hold times on crack growth behavior is not significant. The zero hold times tests are presently in progress. The results to date follow the 0.1 minute and 1 minute tests very closely. For the unirradiated specimens, Figure NRL.2 shows that the hold time tests also produced a negligible effect on crack growth behavior. Comparison of Figures NRL.1 and NRL.2 indicates that the fatigue response of the neutron irradiated and unirradiated specimens is nearly the same when compared on the basis of da/dN. However, a similar comparison on the basis of crack growth rate per unit time, da/dt, shows that the crack growth rates decreased in direct proportion to the length of the hold time employed for both the unirradiated and neutron irradiated specimens.

At equal hold times, the growth rates on a da/dt basis were nearly equal for both irradiated and unirradiated specimen groups at similar values of ΔK . All results indicate that the cyclic rather than the static loading was primarily responsible for the observed crack growth behavior for the neutron irradiated and unirradiated material at 800°F (427°C). Further experiments will examine the hold time effect in neutron irradiated and unirradiated 20% cold-worked Type 316 SS and nickel-base alloys at 1100°F (593°C).

Conclusions

1. The effect of hold time on fatigue crack growth rate, da/dN, as a function of stress intensity factor range, ΔK , for fast neutron irradiated 20% cold-worked Type 316 SS was found to be negligible for postirradiation tests at 800°F (427°C).

2. Similarly, for unirradiated material tests at 800°F (427°C), crack growth behavior was nearly independent of hold time.

3. Comparison of the neutron irradiated and unirradiated fatigue results indicates that neither irradiation nor hold time have an appreciable effect on crack growth rate in 20% cold-worked Type 316 SS for the ΔK range investigated.

B. HIGH FLUENCE ASSESSMENTS OF 0.03% COPPER PLATE FROM 30-TON A533-B DEMONSTRATION MELT

J. R. Hawthorne

Background

The first large scale demonstration test of improved 550°F (288°C) radiation resistance in steel by the minimization of copper, phosphorus, and other impurities was accomplished with a 30-ton commercial melt of A533-B.⁽⁸⁾ The melt composition included 0.03% Cu, 0.008%P,

FIGURE NRL.1. Effect of Hold Time on Fatigue Crack Growth Rates in Neutron Irradiated 20% Cold-Worked Type 316 Stainless Steel as a Function of the Stress Intensity Factor Range.

FIGURE NRL.2. Effect of Hold Time on Fatigue Crack Growth Rates in Unirradiated 20% Cold-Worked Type 316 Stainless Steel as a Function of the Stress Intensity Factor Range.

and 0.008%S compared to melt specification limits of 0.10%Cu max, 0.010%P max, and 0.012%S max. Two ingots were cast and fabricated as 6-in. thick plates. For a special test of impurity composition effects, one half of the melt was modified by a copper addition during ingot casting, i.e., the copper content was increased to 0.13%Cu. Class 1 and Class 2 plates with the primary melt composition were subsequently found to have markedly improved 550°F (288°C) radiation resistance over that of typical commercial production plates with higher impurity levels. Results for the primary versus modified composition plates separately confirmed the particularly detrimental effect of copper on radiation resistance. Building upon these observations, the present study was undertaken to investigate the full potential of the primary melt composition for severe nuclear service applications. The growing interest in the capabilities of improved composition ferritic alloys for high fluence applications in fast breeder and other advanced reactor types was one stimulus for the recent investigation.

Progress

Notch ductility retention after high fluence was explored for the 0.03%Cu Class 1 plate using a series of five irradiation experiments. Charpy-V specimens were exposed at 530, 550, and 600°F (277, 288, and 316°C) to fluences ranging from 1.5 to 6.6×10^{20} n/cm² >1 MeV (calculated neutron spectrum). The irradiation facility employed was a water cooled loop of the Advanced Test Reactor. Tensile specimens were included in one 550°F (288°C) experiment.

Postirradiation Charpy-V results are given in Figure NRL.3. The aggregate data permit several observations, the most important of which is the indication of strong potential for high fluence service of this material at 530 to 600°F (277 to 316°C). A fluence of 2.3×10^{20} n/cm² at 530°F, for example, did not raise the Charpy-V 30 ft-lb transition temperature above 250°F (121°C). Relatively high upper shelf energy retention with irradiation is also indicated. As a second observation, radiation embrittlement saturation is not evident in the data for either 530°F (277°C) or 600°F (316°C) irradiation conditions. The data do suggest, however, that small irradiation temperature differences in the range 530 to 600°F are quite significant to irradiation response at this fluence level. The difference noted in transition temperature increase for 550°F (288°C) versus 600°F (316°C) irradiation conditions, in particular, is somewhat larger than would be expected from observations at a decade lower fluence, i.e., 2 to 3×10^{19} n/cm².

Yield strength values following 2.5×10^{20} n/cm² at 550°F (288°C) were 84.5 ksi at 250°F (121°C), 80.8 ksi at 400°F (204°C), 77.8 ksi at 550°F (288°C). Ratio Analysis Diagram assessments of yield strength and Charpy-V upper shelf energy level for this exposure condition indicate retention of plastic fracture qualities after irradiation, even for thick (6-in.) components.

In summary, the potential of improved composition ferritic structural steels for high fluence advanced reactor applications appears enhanced by the present findings. Further definition of high fluence property trends as a function of exposure temperature would be important, however, to such utilization.

FIGURE NRL.3. Charpy-V Notch Ductility Retention of Plate Section A (Primary Melt Composition) Following High Fluence Exposure at Elevated Temperatures. (A capability for high fluence service is clearly indicated.)

II. RADIATION DAMAGE MECHANISMS

A. EXAMINATION OF MICROSTRUCTURE OF INCONEL 718, INCOLOY 800, PH13-8Mo, MOLYBDENUM AND NIOBIUM AFTER IRRADIATION

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Background

Swelling, or the increase in volume which accompanies the formation of interstitial dislocation loops and voids and the partitioning of point defects between them, is a problem of major engineering and economic importance for advanced reactor systems. The mechanism of irradiation-induced swelling in metals subjected to high fluence, high temperature neutron irradiations is only partially understood at the present time. Among the variables known to influence swelling resistance are major alloy component compositions, small alloy additions, precipitate structures, and dislocation structures. This work is part of an effort to survey the swelling behavior of various alloy systems and pure metals under neutron irradiation.

Progress

Postirradiation examination of the microstructures of specimens of Inconel 718, Incoloy 800, PH13-8Mo, molybdenum and niobium irradiated in EBR-II Pin 65, Subassembly X123, position F91, has been completed during this period. Specimens of these materials were in the form of transmission electron microscopy (TEM) wafers. Space in this position was made available through the courtesy of Hanford Engineering Development Laboratory (HEDL) and irradiation

conditions for the location as furnished by HEDL were 7.4×10^{21} n/cm² >0.1 MeV at a temperature of 540°C (1004°F). These specimens were encapsulated in a 316 SS subcapsule in a helium atmosphere and therefore were not exposed to liquid sodium during the irradiation.

Compositions of the alloys are given in Table NRL.1. The molybdenum and niobium specimens were high purity MARZ grade materials obtained from Materials Research Corporation in the form of foil. Heat treatments for the specimens after rolling to .004-in. thick foil from stocks of the alloys are given in Table NRL.2. The specimens were thinned to transparency by electropolishing and examined with a JEM 200A electron microscope, operating at 200 kV. Void sizes were measured on photographs using a Zeiss particle size analyzer and foil thicknesses were determined from stereo measurements.

TABLE NRL.1

ALLOY COMPOSITION

Material	Ni	Cr	Fe	C	Mn	P	S	Si	Ti	Al	Nb+Ta	Mo
IN 718	53.1	18.3	18.5	.01	.20	.01	.005	.10	.89	.59	5.20	3.06
IN 800	30.5	20.8	47.0	.09	.71	-	-	.61	-	-	-	.27
PH13-8	8.1	12.6	76.0	.041	.01	.002	.002	.02	-	1.08	-	2.18

TABLE NRL.2

HEAT TREATMENT

Inconel 718	1375°F (745°C) for 2-1/2 hrs, air cool; 1150°F (620°C) for 12 hrs
Incoloy 800	2075°F (1135°C) for 1 hr, air cool.
PH13-8	1815°F (990°C) for 1 hr, air cool to room temperature; Age 1050°F (565°C) for 1 hr.
Molybdenum	2600°F (1427°C) for 2 hr.
Niobium	2600°F (1427°C) for 2 hr.

Observations of each material are described and discussed separately.

Inconel 718 is a nickel-base alloy produced by the International Nickel Company (INCO) strengthened by precipitation of γ' , Ni₃ (Al, Ti) and γ'' , Ni₃ (Nb Ta). A very fine dispersion of γ' is produced by the heat treatment used in this experiment and a few larger spherical precipitates believed to be carbides are also present as shown in Figure NRL.4. Irradiation caused a coarsening of the precipitates as shown in Figure NRL.5. Some denuding of precipitates around large carbide particles and along grain boundaries was observable. Voids were found in the Inconel 718 as shown in Figure NRL.6 with a mean diameter of 113Å, density of 1.2×10^{14} cm⁻³ and swelling of .01%. Many of the voids appeared to be associated with small precipitates.

Incoloy 800 is a high nickel austenitic SS produced by Huntington Alloys which is solid solution hardened. The Incoloy 800 prior to irradiation had only a few large precipitates, presumably carbides. Following irradiation, precipitates were observed along deformation bands and voids were frequently associated with these precipitates as shown in Figure NRL.7. Dislocation loops formed profusely during irradiation and appear to be aligned preferentially along certain directions as shown in Figure NRL.8. Mean void diameter was 215Å, void density was 1.0×10^{14} cm⁻³ and swelling was .064%.

FIGURE NRL.4. Microstructure of Inconel 718 Prior to Irradiation Showing Very Fine Precipitates of γ' and γ'' within the Grains and Larger Carbide Particle.

FIGURE NRL.5. Microstructure of Inconel 718 After Irradiation to 7.4×10^{21} n/cm^2 at 540°C (1004°F) Showing Coarsening of γ' and γ'' Precipitates.

FIGURE NRL.6. Higher Magnification Micrograph of Inconel 718 Showing a Few Voids and the Precipitate Structure.

FIGURE NRL.7. Micrograph of Incoloy 800 After Irradiation to a Fluence of 7.4×10^{21} n/cm² at 540°C (1004°F) Showing Voids and Precipitates Formed During Irradiation.

FIGURE NRL.8. Micrograph of Irradiated Incoloy 800 with Dislocation Loop Structure in Contrast. (Compare with Figure NRL.7 which shows void and precipitate structure.)

PH13-8Mo is a martensitic precipitation-hardening SS (maraging) produced by Armco Steel Corporation. The grain morphology is derived from the martensite laths formed during transformation and thus has many long, narrow grains. Tempering at 1050°F (565°C) causes some coarsening of the dislocation structure but a high dislocation density remains. There are a few large blocky precipitates in the grains, probably cementite. No notable changes in the precipitate structure occur during irradiation nor was there any marked change in the dislocation structure so Figure NRL.9 is representative of both the irradiated and unirradiated microstructure in PH13-8Mo except for the presence of a few voids. These voids are quite heterogeneously distributed and not all grains contain voids. Voids are frequently associated with precipitates. Mean void size was 129Å, void density was 7.9×10^{13} and swelling was .014%.

The molybdenum specimen had a void density of $1.2 \times 10^{17} \text{ cm}^{-3}$, a mean diameter of 41Å, and swelling of .47%. Typical microstructures are shown in Figure NRL.10.

The niobium specimen examined had a void density of $3.9 \times 10^{17} \text{ cm}^{-3}$, a mean void diameter of 25Å, and a swelling of .35%. Typical microstructures are shown in Figure NRL.11.

Summary

Microstructures of four classes of materials, a precipitation-hardening nickel base alloy, a solid solution hardened nickel-base alloy, a martensitic alloy, and refractory metals were examined after irradiation to a fluence of $7.4 \times 10^{21} \text{ n/cm}^2$ at 540°C (1004°F). Swelling was low in the alloys and the most significant observation was the coarsening of precipitates in the Inconel 718. Swelling in the refractories was about .4% even though the temperature was less than 0.3 of the absolute melting point.

B. FLUENCE DEPENDENCE OF ION DAMAGE IN NICKEL

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Background

Although the void and dislocation structures produced by neutron and charged particle irradiations of metals are qualitatively similar, the quantitative details of the structures are controlled by a large number of factors, many of which are not yet fully understood. The aims of the present research are to produce a systematic set of data on the effects of ion-irradiation parameters on the void and dislocation structures in pure nickel and to compare these data with the predictions of theoretical models for void nucleation and growth. Previously reported segments of this work have examined the effects of irradiation temperature,⁽⁹⁾ ion flux (dose rate),⁽¹⁰⁾ and irradiation dose at 575°C.⁽¹²⁾ The following part of this report discusses additional dose-dependence results at 575, 625, and 675°C.

Experimental Results

Details of the techniques used for specimen preparation, ion bombardment, observation and analysis have been described previously, so only a brief account of the factors that are specific to the present experiments will be given. The material used was MARZ grade (>99.995%) nickel foil obtained from Materials Research Corporation. The specimens were irradiated with 2.8 MeV Ni^{+} ions at an ion current density of $5 \mu\text{A/cm}^2$, which was calculated⁽¹³⁾ to produce

FIGURE NRL.9. Micrograph of PH13-8Mo, a Maraging Steel, After Irradiation to a Fluence of 7.4×10^{21} n/cm² at 540°C (1104°F). (Note the heterogeneous distribution of voids from grain to grain in this material.)

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FIGURE NRL.10. Micrograph of Pure Molybdenum Irradiated to a Fluence of 7.4×10^{21} n/cm² at 540°C (1004°F) Showing a High Density of 41Å Diameter Voids.

FIGURE NRL.11. Micrograph of Pure Niobium Irradiated to a Fluence of 7.4×10^{21} n/cm² at 540°C (1004°F) Showing a High Density of 25Å Diameter Voids.

a maximum displacement rate of 7×10^{-2} displacements per atom per second (dpa/sec) at a depth of 5000Å from the bombarded surface. Ion fluences were chosen to produce maximum damage levels between 1.3 and 130 dpa, and specimens were irradiated at temperatures of 575, 625, and 675°C. Void and dislocation structures at the depth of maximum damage were studied by transmission electron microscopy (TEM).

The void and dislocation structures observed in specimens irradiated at 575°C to damage levels of 4, 13, 40 and 130 dpa were discussed in a previous report.⁽¹²⁾ Some of these data will be repeated, however, since they are relevant to the more complete analysis presented in this report. The microstructures of all the specimens irradiated between 575 and 675°C contained voids and dislocations. Void distributions that were suitable for quantitative analysis were observed in all of the specimens irradiated at 575°C (1.3, 4, 13, 40, 80, and 130 dpa), and in the specimens irradiated to 1.3, 4, 13, and 40 dpa at 625°C. The specimens irradiated to 130 dpa at 625 and 675°C both contained a number of very large voids, >2000Å in diameter as shown in Figure NRL.12 for the 675°C specimen. These made it difficult to determine effective damage levels for these specimens, since the relatively constant portion of the damage versus depth relationship was also approximately 2000Å wide. The void populations observed in the specimens irradiated to 1.3 and 4 dpa at 675°C were very heterogeneous, which made meaningful void measurements impossible. An example of the heterogeneity observed in the 4 dpa, 675°C specimen is shown in Figure NRL.13. Quantitative descriptions of the void and dislocation structures in the specimens irradiated at 575 and 625°C are given in Table NRL.3.

TABLE NRL.3
VOID AND DISLOCATION DISTRIBUTIONS IN
NICKEL-ION-IRRADIATED NICKEL

Irrad. Temp. (°C)	Irrad. Dose (dpa)	Void Density (cm ³)	Mean Void Diameter (Å)	Swelling (%)	Disl. Density (cm)
575	1.3	4.7×10^{15}	85	0.16	not meas.
575	4	1.1×10^{16}	75	0.28	2.7×10^{10}
575	13	6.9×10^{15}	130	0.90	2.0×10^{10}
575	40	5.5×10^{15}	150	1.23	2.4×10^{10}
575	80	2.0×10^{15}	255	2.4	1.5×10^{10}
575	130	6.6×10^{14}	380	2.2	7.8×10^9
625	1.3	1.0×10^{15}	125	0.13	not meas.
625	4	7.0×10^{14}	230	0.50	8.6×10^9
625	13	1.8×10^{15}	225	1.2	5.6×10^9
625	40	2.7×10^{14}	550	2.6	4.7×10^9

As was done previously for the temperature and dose rate dependence data at 13 dpa and the initial fluence dependence at 575°C, the swelling behavior of these specimens was compared with the predictions of the Brailsford and Bullough model⁽¹⁴⁾ for the swelling rate due to void growth. Since the formulae used in these calculations have been presented previously,^{(9),(12)} they will not be repeated here. The main ideas of the model, as they apply to the system of nickel-ion irradiated nickel, are the following: the irradiation produces equal

FIGURE NRL.12. Voids Observed in High-Purity Nickel, Following Irradiation at 675°C with 2.8 MeV $^{58}\text{Ni}^+$ Ions to a Damage Level of 130 dpa.

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FIGURE NRL.13. Heterogeneous Distribution of Voids Observed in High-Purity Nickel, Following Irradiation at 675°C with 2.8 MeV $^{58}\text{Ni}^+$ Ions to a Damage Level of 4 dpa.

numbers of vacancies and interstitials. These point defects, which are mobile at the temperatures of interest, are removed from the material either by mutual recombination or by migration to sinks, which consist of voids and dislocations. The voids represent neutral sinks, equally strong for both vacancies and interstitials. The dislocations, due to a stronger strain interaction with the interstitials, are biased sinks, having a stronger attraction for interstitials than for vacancies. This bias causes a net flow of interstitials to dislocations and a net flow of vacancies to voids, resulting in void growth. If the void and dislocation parameters, as given in Table NRL.3, are known, the principal undetermined factor in the model is the bias of the dislocations for interstitial capture which has been assumed to be 1 to 2% by several investigators.¹⁴

As reported previously, the specimens irradiated at 575°C exhibited rapid swelling up to 13 dpa, followed by swelling at a much lower rate above that damage level. Similar behavior has been reported for 525°C nickel-ion irradiation of nickel by Ryan and Taylor.⁽¹⁵⁾ The comparison of the present 575°C data and the Brailsford-Bullough model is illustrated in Figure NRL.14. The dashed line below 13 dpa represents the swelling rate calculated using

FIGURE NRL.14. Comparison of the Experimental Fluence Dependence of Swelling Observed in Nickel at 575°C (data points) and the Swelling Rates Calculated by the Void Growth Model (dashed and solid lines). (See text for the details of the calculations.)

the 4 dpa void and dislocation parameters and a 6% bias for interstitial capture by dislocations. The solid curve above 13 dpa is the swelling calculated with the model for a 1.4% bias. This calculation was performed in the following way: the microstructure parameters at each experimentally investigated dose level were used to calculate a swelling rate for that dose level; the swelling rates for the dose levels between the experimental points were determined by linear interpolation between these calculated rates; the swelling was calculated by integrating these rates, using the measured swelling at 13 dpa as the initial value. As can be seen from the figure, the agreement between the void growth model and the experimentally measured swelling was quite acceptable. A possible explanation for the 6% bias required to match the swelling rate below 13 dpa to the model, involving the change of the character of the dislocation structure from loops at lower doses to an interconnected network at higher doses, has been discussed previously.⁽¹²⁾

When the Brailsford-Bullough void growth model was applied to the 625°C irradiations, as shown in Figure NRL.15, the agreement was not nearly as good. The data points and solid line represent the experimentally measured swelling, and the dashed line represents the swelling rate calculated by the void growth model using the 13 dpa void and dislocation parameters and a 1.5% bias for interstitial capture by dislocations. To match the experimentally measured swelling rate, in fact, it was necessary to use approximately a 10% bias. It seems unlikely that the dislocation bias would change this radically between 575 and 625°C, since the structures observed for both temperatures at 13 dpa,⁽⁹⁾ as well as the other dose levels investigated at 625°C, consisted of an interconnected network of dislocations. It therefore appears that some factor affecting the swelling rate in these experiments is not being properly considered by the form of the void growth theory applied here. A possible source of this disagreement is the model used to calculate the strengths of the void and dislocation structures as sinks for point defects. For the high displacement rate of these experiments, the sink strengths very strongly affect the magnitudes of calculated swelling rates, so other models for point defect loss to sinks, such as the surface-reaction-limited model of Mansur, et al⁽¹⁶⁾ may provide a better fit to the experimental swelling data. Additional analysis and some additional experimental data will be required to test these possibilities.

Conclusions

Comparison of the fluence dependence of swelling in nickel-ion irradiated nickel with the predictions of the Brailsford-Bullough model⁽¹⁴⁾ for the swelling rate due to void growth has indicated the following:

1. At 575°C, the observed swelling above 13 dpa agreed well with the swelling integrated from the calculated swelling rates of the model, using a dislocation bias for interstitial capture of ~1.4%.
2. At 625°C, however, it was not possible to use the model with the bias determined at 575°C to calculate swelling rates matching the experimentally observed ones. It appears that further development of the model will be necessary if it is to have this type of predictive capability.

FIGURE NRL.15. Comparison of the Experimental Fluence Dependence of Swelling Observed in Nickel at 625°C (data points, solid line) and the Swelling Rate Calculated by the Void Growth Model (dashed line), Using the 13 dpa Microstructure Parameters and a 1.5% Dislocation Bias for Interstitial Capture.

C. ION-SIMULATED IRRADIATION-INDUCED CREEP OF REACTOR STRUCTURAL ALLOYS

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Background

There has been a growing need in this country and throughout the world to accurately predict the deformation arising from the irradiation-induced creep occurring in the core structural components of advanced breeder reactors currently under development. This need has generated considerable interest in possible techniques for simulating irradiation-induced creep using particle accelerators thereby alleviating the need of conducting costly and time-consuming in-reactor tests. Additional benefits accrue from the ability to screen potential alloys and to carefully control experimental parameters such as temperature, stress, and flux.

An experimental apparatus has been constructed at NRL for the purpose of measuring the ion-simulated irradiation-induced creep rate of potential fast breeder reactor structural materials. During the current reporting period, progress in the development of this apparatus has been made including installation at the NRL Cyclotron and a number of preliminary irradiation tests. These tests have served to highlight problem areas and allow necessary modifications to be made to the experimental apparatus.

Progress

The installation of the ion-simulated irradiation-induced creep apparatus in experimental room number two of the NRL Sector Focusing Cyclotron is shown schematically in Figure NRL.16. An elevated view of the beam line and its components is shown in Figure NRL.17. Following the installation of the apparatus and alignment of the beam line, a series of irradiation tests was conducted using 70 MeV alpha particles. These tests were invaluable in assessing the validity of various calculations made and confirming expectations based upon previous proton-simulated irradiation-induced creep studies.⁽¹⁸⁾ For example, it was demonstrated that air cooling of the heat sink was adequate. However, the maximum beam current obtained to date is approximately one half that previously anticipated. Therefore, future tests will be necessary in order to optimize techniques for the efficient extraction of circulating beam. Reduced beam current inevitably results in increased irradiation time.

As a result of the first two irradiation tests, modifications to the wire profile scanner originally developed by the Cyclotron Branch⁽¹⁹⁾ were refined, permitting on-line profile monitoring at maximum expected beam current levels. These modifications included the addition of 0.23 cm (.090 in.) diameter tungsten scanning wires and ceramic insulators. The successful operation of the profile scanner made possible the judicious selection of specimen gauge length and width. Based upon these initial tests, it was determined that a reasonably uniform beam could be obtained over a vertical height of approximately 1.25 cm (0.5 in.).

A parallel effort has been underway to develop an online data acquisition and processing system using the Cyclotron's Sperry Univac Series 60 computer. It is anticipated that the data processing system will provide continuous monitoring of critical parameters during irradiation creep tests and permit early recognition of abnormal occurrences that may result in loss of strain resolution or otherwise jeopardize the data acquisition during test operations.

FIGURE NRL.17. Irradiation-Induced Creep Experimental Beam Line.

Succeeding irradiation tests were used primarily to ascertain strain resolution under actual operating conditions and focus attention on modifications needed to achieve a strain resolution of at least 10^{-5} in./in. Temperature regulation of the specimen and associated load train components has been identified as a problem area requiring further development. As described elsewhere^{(17),(19)} the instantaneous strain is extremely sensitive to thermal expansion caused by temperature fluctuations in the specimen, grips, pullrods, and associated components. In an effort to further reduce this thermal expansion, a heat sink temperature control system was developed, in addition to the previously existing specimen temperature control system. Although further development in this area is necessary, considerable progress has been made. The ultimate objective is to maintain the desired strain resolution during anticipated beam current fluctuations.

In an effort to reduce anomalously high specimen gradients, the most recent irradiation tests have been conducted in a nitrogen environment. Nitrogen was chosen initially due to its compatibility with the Vac Ion pumping system. It was discovered that although surface gradients were reduced dramatically, the response of the infrared heater was considerably slower due to the cooling obtained by the nitrogen environment.

Future Plans

Once the ion-simulated irradiation-induced creep experimental apparatus has been successfully developed, a short series of irradiation tests using pure nickel specimens will be conducted. Nickel has been shown to exhibit an anomalously high irradiation-induced creep rate.^{(17),(20),(22)} Following the demonstration of irradiation-induced creep in nickel, a series of tests using AISI Type 316 SS is planned. These tests will be conducted for the purpose of measuring both the transient and steady state irradiation-induced creep rate as a function of stress, temperature, and flux. In addition, electron microscopy studies of both irradiated and control specimens will be used along with the creep data obtained to develop consistent creep mechanism theories. Finally, computer modeling will be necessary to determine radiation damage and allow correlation with both current and future fast neutron irradiation data.

III. ARMY REACTOR STUDIES

A. NOTCH DUCTILITY AND STRENGTH OF TYPE 316 STAINLESS STEEL SUBMERGED ARC WELD DEPOSITS

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Background

Submerged arc girth welds join the upper nozzle and flange assembly and the lower head of the MH-1A reactor vessel to the central vessel forging surrounding the fuel core. The welds, because of their proximity to the fuel core, will receive some radiation exposure in service. The susceptibility of the weld deposits to radiation embrittlement and the level of postirradiation notch toughness are, therefore, of interest to Army sponsors.

Archive weld metal for radiation effects studies was not secured at the time of vessel manufacture. Accordingly, an experimental 1-in. thick test weld (AISI Type 316) was made for exploratory evaluations. Submerged arc welding conditions and parameters were selected to be

representative of one portion of the 3-1/8-in. thick vessel welds (root region of the double U joint). Vessel fabrication records showed only weld specification ranges and not actual welding parameters; therefore average values within each range were used.

Progress

An assessment of weld deposit notch toughness before and after 500°F (260°C) irradiation to $4.6 \times 10^{19} \text{ n/cm}^2 > 1 \text{ MeV}$ was presented in the previous report of progress.⁽²³⁾ Charpy-V values for pre- and postirradiation conditions were found to be much lower than that of the vessel forging. Moreover, the Charpy-V energy level after irradiation was only 35 ft-lb. In view of this observation, Army sponsors requested a more detailed study of the effects of welding parameters and conditions on notch toughness and radiation resistance. For this follow-on study, three additional submerged arc test welds were made using dcrp welding conditions. A fourth weld with ac conditions was also made. Welding details and materials are documented in Table NRL.4. Note that in addition to welding parameter variations, more than one weld filler and flux combination was used to explore the influence of lot-to-lot variations on properties of the three dcrp welds. Weld V23 is believed best representative of the second portion of the actual MH-1A vessel welds (non-root region). Chemical compositions of the weld deposits are not yet available.

Tensile strength determinations for the dcrp welds have been completed and are compared in Table 5. Tensile tests of the ac weld (Code S85) are still in progress. As indicated, the yield strengths of welds V23, V24, and V25 were approximately equal at 75°F (24°C) and at 500°F (260°C) and were approximately 5 ksi higher than those of the original dcrp test weld S83. The tensile strengths of the four welds were comparable, however. In general, the differences in individual welding parameters, conditions or materials used did not result in an appreciable variation in weld deposit strengths.

Significant differences in Charpy-V notch ductility were found among the welds (Table 6). The Charpy-V energy level of weld V23, in particular, is seen to be higher than that of weld V24 at 75°F (24°C) and at 500°F (260°C). This comparison suggests that Charpy-V energy level for this type of weld is dependent on the welding parameters used; welding materials were the same for both welds (Table NRL.4). Data for welds S83 versus S85, on the other hand, suggest that the choice of welding conditions, i.e., dcrp versus ac, is not critical to Charpy-V energy level for this type of weld. The data for welds V24 versus V25 are also comparable; accordingly, the difference in welding flux lot was not significant in this case. Limited data for 500°F (260°C)-1168 hour thermal conditioning of weld S85 are also shown in Table NRL.6. The conditioning treatment would appear to increase Charpy-V energy level slightly for a 500°F (260°C) test temperature but not for 900°F (482°C) or 1100°F (593°C) test temperatures.

A 500°F (260°C) radiation assessment of two of the weld deposits is planned for the next reporting period, together with the development of additional data for weld V24 (unirradiated condition).

TABLE NRL.4

WELDING PARAMETERS AND CONDITIONS FOR AISI TYPE 316 SUBMERGED ARC TEST WELDS
(SIMULATED ARMY MH-1A REACTOR WELD INVESTIGATIONS)

Parameter/ Condition	Weld Identification Code				
	V23	V24	V25	S83	S85
Base plate	a	a	a	a	a
Weld procedure	b	b	b	b	b
Filler type	Type 316 ^c (5/32-in. diam)	Type 316 ^c (5/32-in. diam)	Type 316 ^c (5/32-in. diam)	Type 316 ^d (1/8-in. diam)	Type 316 ^d (1/8-in. diam)
Flux	Arcos S-16 (lot 3K1F)	Arcos S-16 (lot 3K1F)	Arcos S-16 (lot 2F4F)	Arcos S-16 (lot 2F4F)	Arcos S-16 (lot 2F4F)
Backing	Type 316 (5/16 x 1-in.)	Type 304 (1/4 x 1-in.)	Type 304 (1/4 x 1-in.)	Type 316 (1/4 x 1-in.)	Type 316 (1/4 x 1-in.)
Joint	e	e	e	f	f
Restraint	Full, Mech.	Full, Mech.	Full, Mech.	Full, Mech.	Full, Mech.
Current	500A, dcrp	420A, dcrp	420A, dcrp	420A, dcrp	420A, ac
Voltage	32V	30V	30V	30-32V	30-32V
Travel	17 ipm	12 ipm ^g	12 ipm ^g	12 ipm ^g	12 ipm ^g
Stickout	1-1/4 in.	1-1/4 in.	1-1/4 in.	1-1/4 in.	1-1/4 in.
Torch Angle	90 deg	90 deg	90 deg	90 deg	90 deg
Interpass (°F/max)	300	300	300	300	350
Interpass Cleaning	h	h	h	h	h
Postweld HT	none	none	none	none	none
Inspection	-	-	-	X-ray	X-ray

a - AISI Type 316 plate (1-in.), A1 heat 65808

b - Submerged arc (S/A), single electrode

c - Arcos lot E2061R316

d - Unibraze heat 13811120

e - Single Vee (1/4-in. root opening; 90 deg incl. angle)

f - Single Vee (1/8-in. root opening; 90 deg incl. angle)

g - root pass was at 16 ipm

h - acetone, wire brush

TABLE NRL.5

TENSILE PROPERTIES OF AISI TYPE 316 SUBMERGED ARC
TEST WELDS (UNIRRADIATED CONDITION)
(Simulated Army MH-1A Reactor Weld Investigations)

Weld Code	Thickness (in.)	Temperature (°F) (°C)		Yield Strength ^a (ksi)	Yield Strength ^b (ksi)
V23	1	75 500	24 260	65.3 50.8	88.3 72.3
V24	1	75	24	65.8	90.1
V25	1	75 500	24 260	65.8 50.6	87.5 ^c 70.8 ^c
S83	1	75 500	24 260	60.1 44.6	87.0 ^c 68.3 ^c

^a 0.226-in. diam x 1.125-in. gage length specimens.

^b average of duplicate tests except where noted.

^c single test determination.

TABLE NRL.6

CHARPY-V NOTCH DUCTILITY OF AISI TYPE 316 SUBMERGED ARC
TEST WELDS (UNIRRADIATED CONDITION)
(Simulated Army MH-1A Reactor Weld Investigations)

Weld Code	Charpy-V Energy (ft-lb)				
	-100°F (-73°C)	75°F (24°C)	500°F (260°C)	900°F (482°C)	1100°F (593°C)
V23	-	70 70 63	81 84	80	72
V24	-	55 56 57	66 74	-	-
V25	-	57	75	59	58
S83	-	52	58		
S85 (ac weld)	43	50 54	55 50	65	48
S85 (thermally conditioned) (500°F-1168 hr)	-	-	60 61	45 49	49 50

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I. LIQUID METAL FAST BREEDER REACTOR PROGRAM

A. EFFECTS OF PREIRRADIATION MICROSTRUCTURE ON SWELLING AND MECHANICAL PROPERTIES

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Purpose

Irradiation-induced swelling and loss of ductility are two major problems in the use of Type 316 stainless steel as a fuel cladding and core structural alloy in LMFBs. 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 or not the postirradiation ductility can be improved by control of the preirradiation microstructure while still maintaining swelling resistance through a high sink density.

Experiment Description

Samples of Type 316 stainless steel were given various combinations of cold-working and thermal aging treatment before irradiation in EBR-II subassemblies X100 and X100A. Most of the samples were irradiated at 500 and 600°C although for some heat treatment conditions irradiation temperatures of 400, 450, and 700°C were also included. The maximum fluence was 7.8×10^{22} n/m² (>0.1 MeV).

Results

Preliminary results of immersion density measurements, transmission electron microscopy, and postirradiation creep-rupture tests on samples irradiated in subassemblies X100 and X100A were given in the previous report.⁽¹⁾⁽²⁾ During the present reporting period transmission microscopy on samples from subassembly X100A was continued.

Microstructural Analysis

A summary of the available results from transmission electron microscopy examination of the irradiated samples is given in Table ORNL.1. A detailed description of the various microstructures follows.

Type 316 Stainless Steel Solution Annealed for 1 hr at 1050°C

The variation in microstructure in annealed Type 316 stainless steel as a function of fluence and irradiation temperature is shown in Figure ORNL.1. In all cases the voids appeared to be evenly distributed throughout the material. Many of the voids present in the specimen irradiated to a fluence of 3.0×10^{22} n/cm² at 500-520°C appeared to be associated with precipitate particles.

*Research sponsored by the Energy Research and Development Administration under contract with the Union Carbide Corporation.

TABLE ORNL.1

SUMMARY OF THE EFFECTS OF PREIRRADIATION HEAT TREATMENT ON THE POSTIRRADIATION
MICROSTRUCTURE OF TYPE 316 STAINLESS STEEL

Preirradiation Heat Treatment	Irradiation Conditions		Immersion Density Change (%)	Concentration of Voids (cm ⁻³)	Mean Void Diameter (Å)	Microstructural Observations
	Temperature (°C)	Fluence >0.1 MeV (n/cm ²)				
Annealed 1 hr at 1050°C	500-520	3.0 x 10 ²²	+0.14	1.1 x 10 ¹³	498	Larger voids were usually associated with precipitates. At 600-660°C and 6.6 x 10 ²² n/cm ² (>0.1 MeV), a bimodal distribution was present. Large voids were on precipitates and small voids in the matrix.
	500-535	5.6	-0.37	4.9	485	
	600-615	3.0	+0.23	NA	NA	
	600-660	6.6	-1.53	0.63 (Matrix 0.26)	1365 (Matrix 937)	
Annealed 1 hr at 1050°C +20% Cold-Work	500-555	6.6	+0.10		400-600	Distribution of voids was inhomogeneous with most voids located between bands in areas where recovery was well advanced. In addition the number of voids in the specimen irradiated at 600-660°C was approximately a factor of 2 higher than in the specimen irradiated at 500-555°C.
	600-660	6.6	-0.40		600-1500	
Annealed 1 hr at 1050°C + 20% Cold-Work + 10 hr at 650°C	450	7.8	-0.35		~200-400	For specimens irradiated at 450 or 500°C, voids were located between deformation bands in areas where recovery was well advanced. Precipitates were preferentially associated with grain boundaries and deformation bands. Voids were also aligned parallel to these bands at 700°C and 7.8 x 10 ²² n/cm ² . The structure was completely recovered with second phase particles aligned along boundaries together with some small gas bubbles (<100 Å). No voids were seen.
	500	7.8	-0.48		~400-800	
	700	7.8	-0.06		Gas Bubbles <100	
Annealed 1 hr at 1050°C + 20% Cold-Work + 10 hr at 550°C	500	6.6	-0.10		400-1000	Voids located in recovered areas between deformation bands. The number of voids was about 20% as large as in the specimen aged at 650°C.

FIGURE ORNL.1. Microstructures of Annealed Type 316 Stainless Steel Irradiated at About 500 and 600°C. (a) 3.0×10^{22} n/cm², 500-520°C $\Delta\rho/\rho = +0.14\%$. (b) 5.6×10^{22} n/cm², 500-535°C $\Delta\rho/\rho = 0.37\%$. (c) 6.6×10^{22} n/cm², 600-660°C $\Delta\rho/\rho = 1.53\%$.

Increasing the fluence to 5.6×10^{22} n/cm² at 500-535°C did not seem to affect the void distribution significantly. However, there were some noticeable changes. The number of voids was slightly larger while the average size (~ 550 Å) was approximately the same as in the lower fluence specimen. Again, as in the case of the previous specimen, many of the voids were associated with precipitate particles. In addition to these voids there were a number of smaller voids distributed in the areas between the precipitates.

The final specimen in this series was irradiated to a fluence of 6.6×10^{22} n/cm² at 600-660°C. Immersion density measurements of this specimen showed that the density change was significantly larger than the previous two specimens, although the concentration of voids was somewhat lower than the specimen irradiated at 500-535°C. There were also a large number of precipitate particles present. Examination of this microstructure using stereomicroscopy revealed that these particles were sheet-like in form and had a complex orientation relationship with the matrix. In addition the stereomicroscopy revealed a bimodal void distribution in which all of the large voids (~ 2000 Å average diameter) were associated with the sheet-like precipitate while all of the small voids (average diameter ~ 600 Å) were free of precipitates.

Type 316 Stainless Steel Solution Annealed 1 hr at 1050°C and Cold-Worked 20%

The microstructures of 20% cold-worked Type 316 stainless steel irradiated to a fluence of 6.6×10^{22} n/cm² at approximately 500 and 600°C are shown in Figure ORNL.2. In general the structures were composed of bands of highly deformed material together with areas that were partially recovered. There is also a preferential alignment of precipitates along these bands as well as along grain boundaries.

In particular, the microstructure of the specimen irradiated at 500-550°C was characterized by the banded structure and partially recovered areas that were still heavily dislocated. The distribution of voids was inhomogeneous with most of them located between bands in areas where recovery was well advanced. The average diameter of these voids was 400-600 Å.

In comparison, the structure of the specimen irradiated at 600-660°C appeared to be more recovered, although the banded structure still persisted. It is also of interest to note that the concentration of precipitate particles observed in this specimen was higher both along the bands and in areas between the bands than was seen in the specimen irradiated at 500-550°C. In addition, the number of voids in the specimen irradiated at 600-660°C was approximately a factor of 2 to 3 higher, while the void sizes range from about 600 Å to about 1500 Å. There was also evidence that some of the larger voids were associated with the precipitate particles. The voids were located in the recovered areas between the bands and tended to be aligned parallel to the bands.

Type 316 Stainless Steel, Solution Annealed for 1 hr at 1050°C, Cold-Worked 20% and Aged for 10 hr at 650°C

The microstructures of specimens with a preirradiation heat treatment of 20% cold-work followed by 10 hr at 650°C are shown in Figures ORNL.3 and ORNL.4. These specimens were irradiated to a fluence of $6.6-7.8 \times 10^{22}$ n/cm² at temperatures of 450, 500, or 700°C. A number of obvious differences can be seen in these microstructures. Voids can be seen in the specimens irradiated at 450 and 500°C, while only a few very small gas bubbles could be detected at the

FIGURE ORNL.2. Microstructures of 20% Cold-Worked Type 316 Stainless Steel Irradiated at About 500 and 600°C. (a and b) Irradiated at 500-555°C to 6.6×10^{22} n/cm²; $\Delta\rho/\rho = +0.10\%$. (c and d) Irradiated at 600-660°C to 6.6×10^{22} n/cm²; $\Delta\rho/\rho = -0.40\%$.

$7.8 \times 10^{22} \text{ n/cm}^2$, 450°C
 $\Delta\rho/\rho = -0.35\%$

$6.6 \times 10^{22} \text{ n/cm}^2$, 500°C
 $\Delta\rho/\rho = -0.48\%$

FIGURE ORNL.3. Microstructures of Type 316 Stainless Steel Annealed for 1 hr at 1050°C, Cold-Worked 20%, and Aged 10 hr at 650°C Prior to Irradiation at 450 and 500°C to $6.6\text{--}7.8 \times 10^{22} \text{ n/cm}^2$.

$7.8 \times 10^{22} \text{ n/cm}^2$, 700°C
 $\Delta\rho/\rho = -0.06\%$

FIGURE ORNL.4. Microstructures of Type 316 Stainless Steel Annealed for 1 hr at 1050°C, Cold-Worked 20%, and Aged 10 hr at 650°C Prior to Irradiation at 700°C to $6.6\text{-}7.8 \times 10^{22} \text{ n/cm}^2$.

grain boundaries in the specimen irradiated at 700°C. This observation is consistent with the immersion density measurements, which indicated a maximum swelling in the neighborhood of ~500°C reported in the last reporting period. It should be noted, however, that the voids seen in the specimen irradiated at 450°C were, in general, of much smaller size (~200-400 Å) than those detected in the specimen irradiated at 500°C (~400-800 Å). Samples with identical preirradiation heat treatments irradiated to fluences of 2.5 to 3.0×10^{22} n/cm² showed no evidence of voids and exhibited relatively small density changes ($\leq 0.2\%$) over a similar range of irradiation temperatures.⁽¹⁾

The variation of microstructure with irradiation temperature was also very obvious. The specimen irradiated at 450°C was characterized by deformation bands together with areas that were partially recovered. In addition, precipitates appeared to be preferentially associated with the grain boundaries and deformation bands. Although the voids were not uniformly distributed, they did not seem to be definitely associated with any particular feature, and they appeared to be generally aligned parallel to the banded structure and located between the bands. In this respect the structure was similar to the structure described in specimens that were only cold-worked before irradiation.

The specimen irradiated at 500°C was somewhat more recovered, although dislocations were still present in large numbers within these recovered areas. As in the previous case, there seemed to be some alignment of precipitates parallel to the deformation bands and along grain boundaries. In a few cases voids seemed to be associated with these precipitates. In general, most of the voids were located in the recovered areas between the bands.

The structure of the specimens irradiated at 700°C was very much different. In general the structure was completely recovered with large second-phase particles aligned along grain boundaries. No evidence of voids was seen although in a number of instances what appeared to be small gas bubbles (< 100 Å) could be seen at the boundaries. The density change in this specimen was negligible ($\leq 0.1\%$).

Type 316 Stainless Steel, Solution Annealed for 1 hr at 1050°C,
Cold-Worked 20%, and Aged for 10 hr at 550°C

By way of comparison the microstructure of a specimen given a preirradiation heat treatment of 10 hr at 550°C after annealing and cold-working is shown in Figure ORNL-5. The specimen was irradiated to a fluence of 6.6×10^{22} n/cm² at 500°C. The main features were a partially recovered structure in which large numbers of precipitates were associated with grain boundaries and deformation bands. The recovery was not as advanced as it was in the specimen aged 10 hr at 650°C before irradiation at 500°C. In addition only a small number of voids were present (about 20% as many as in the specimen aged at 650°C), and these were somewhat larger (~400-1000 Å). These voids were located in areas that were completely recovered. The immersion density change measured on this specimen was very low.

FIGURE ORNL, 5. Microstructures of Type 316 Stainless Steel Annealed for 1 hr at 1050°C, Cold-Worked 20%, and Aged 10 hr at 550°C Prior to Irradiation at 500°C to $6.6 \times 10^{22} \text{ n/cm}^2$; $\Delta\rho/\rho = -0.10\%$.

Further Work

Further conclusions as to the effectiveness of various preirradiation treatments will require more detailed analysis and will be reported at a later time. In particular, a more quantitative analysis of void distribution and sizes, dislocation density, and precipitate structures are under way. Comparison of this information with mechanical properties data will provide a basis for determining the optimum preirradiation microstructure for maintaining swelling resistance as well as improving the postirradiation ductility.

B. IN-REACTOR DEFORMATION AND FRACTURE STUDIES

E. E. Bloom

Purpose

The purpose of this experiment is to investigate the in-reactor deformation and fracture behavior of austenitic stainless steels. Specific areas being addressed are: (1) determination of the stress dependency of in-reactor deformation of 20% cold-worked Type 316 Stainless Steel tubing, (2) investigation of the effects of strain accumulated in-reactor on the postirradiation rupture life and ductility, (3) determination of in-reactor fracture strain limits for previously irradiated (EBR-II thimble material) Type 304 Stainless Steel, (4) determination of in-reactor fracture strain limits for 20% cold-worked Type 316 Stainless Steel, and (5) determination of radiation creep mechanisms by transmission electron microscopy of uniaxially stressed specimens.

Experiment Description

The majority of the samples described in this section were irradiated in EBR-II sub-assembly X175A and received an exposure of 8983 MWD. The calculated centerline fluence was $2.5 \times 10^{22} \text{ n/cm}^2$ ($>0.1 \text{ MeV}$). The following is a brief summary of the samples that were contained in the experiment:

- (a) Internally pressurized tubes of 20% cold-worked Type 316 stainless steel were irradiated in holders shown schematically in Figure ORNL.6. Irradiation temperature was calculated to be 550°C and the fluence at the center of the pressurized tube is estimated to be $2.4 \times 10^{22} \text{ n/cm}^2$ ($>0.1 \text{ MeV}$).
- (b) Specimens of Type 304 Stainless Steel from EBR-II safety rod thimbles that had been previously irradiated to about $1 \times 10^{23} \text{ n/cm}^2$ ($>0.1 \text{ MeV}$) were irradiated in holders shown schematically in Figure ORNL.7. These samples were strained in tension during irradiation by pinning them to the specimen holder, which was constructed of high-purity Type 316 stainless steel. The high-purity stainless steel has a swelling rate of 2-3% density decrease per $1 \times 10^{22} \text{ n/cm}^2$ ($>0.1 \text{ MeV}$). It was anticipated that the holder would swell at a faster rate than the samples, thus causing the samples to be stressed and deformed during irradiation. In-reactor fracture strain limits and fracture mode would thus be determined.
- (c) Three specimens of 20% cold-worked Type 316 stainless steel were strained during irradiation in a manner similar to that described under (b) above. The holder body was constructed of annealed high-purity Type 316 stainless steel. The assembly consisted of a rod specimen with threaded shoulders, a holder body, threaded end fixtures, and a sodium-filled subcapsule. The specimen was

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FIGURE ORNL.6. Drawing of the Specimen Holder Used for Irradiation of Internally Pressurized Tubes.

FIGURE ORNL.7. Drawing of the Specimen Holder Used for In-Reactor Fracture Strain Studies. (The holder body was constructed of high-purity Type 316 stainless steel that swelled at a faster rate thus straining the test specimen during irradiation.)

assembled into the holder and the end fixtures tightened to remove all slack but not enough to apply a stress to the specimen. The specimen was stressed during irradiation by differential swelling between it and the holder.

- (d) One sample of annealed Type 316 stainless steel was irradiated in a holder similar to that described in (c) above. Prior to irradiation it was stressed to 10,000 psi (as determined by strain gages that were attached to the sample). Post-irradiation transmission electron microscopy will be used to determine the population of faulted dislocation loops on the various {111} planes. If a larger than random fraction of the loop population lies on a {111} plane normal to the applied stress, this will be an indication that loop alignment is an operative radiation creep mechanism. Annealed rather than cold-worked material was used since in cold-worked material the high initial dislocation density makes identification of the irradiation-produced dislocations difficult.

A few pressurized tube samples were also irradiated in EBR-II subassembly X100A at a calculated temperature of 450°C to an estimated fluence of $2.3\text{--}2.9 \times 10^{22} \text{ n/cm}^2$ (>0.1 MeV). Results from these samples will be included.

Status of Postirradiation Examination

Holders containing pressurized tubes of 20% cold-worked Type 316 stainless steel and uniaxially stressed samples of Type 304 stainless steel from previously irradiated EBR-II thimbles have been disassembled and are presently being examined.

Results of diameter measurements on the twelve pressurized tubes of 20% cold-worked 316 stainless steel irradiated at 550°C to $2.4 \times 10^{22} \text{ n/cm}^2$ (>0.1 MeV) are given in Table ORNL.2. Each value is the average of 16 measurements taken at 1/4-in. intervals over the

TABLE ORNL.2

RESULTS OF DIAMETER MEASUREMENTS ON 20% COLD-WORKED TYPE 316
STAINLESS STEEL TUBES IRRADIATED AT 550°C TO $2.4 \times 10^{22} \text{ n/cm}^2$ (>0.1 MeV)

Sample	Stress (psi)	Mean Diameter (in.)	Diameter Change (%)
3	0	0.2298	-0.09
6	0	0.2298	-0.09
8	0	0.2300	0.00
12	0	0.2298	-0.09
5	10,000	0.2302	+0.09
10	10,000	0.2302	+0.09
1	20,000	0.2309	0.39
4	20,000	0.2311	0.48
9	20,000	0.2310	0.43
11	20,000	0.2310	0.43
7	30,000	0.2317	0.74
2	40,000	0.2302 ^b	

^aAverage of 16 measurements. Measurements taken at 1/4-in. intervals over center 1 in. of a 4-in. sample at 0, 45, 90, 135°.

^bTube apparently depressurized.

center 1 in. of the sample at 0, 45, 90, and 135°. The sample that was to be irradiated with a hoop stress of 40,000 psi apparently depressurized, since the diameter change was much smaller than found in the sample irradiated at 30,000 psi. Attempts to find the leak by pressurizing the tube with helium and watching for bubbles when submerged in alcohol were unsuccessful. It is suspected that the leak was in the seal weld on the capillary fill tube that was removed for the above mentioned pressurization test. Diameter changes are plotted and compared to values calculated from the proposed interim irradiation creep equation in Figure ORNL.8. When adjusted for the densification that occurs in the cold-worked material,

FIGURE ORNL.8. Observed In-Reactor Deformation of 20% Cold-Worked Type 316 Stainless Steel Compared to Present Irradiation Creep Equation.

the irradiation creep correlation underestimates the observed in-reactor deformation by a factor of 4 at a stress of 20,000 psi. Diameter measurements on samples irradiated at 450°C to fluences in the range $2.3\text{--}2.9 \times 10^{22} \text{ n/cm}^2$ ($>0.1 \text{ MeV}$) are listed in Table ORNL.3. The diametral strains observed in the 20% cold-worked samples irradiated at 20,000 psi hoop stress were in better agreement with the strains calculated from the irradiation creep equation than for the tubes irradiated at 550°C. The calculated strain was about 0.16% compared to an observed strain of 0.22% (based on an initial tube diameter of 0.2300) or 0.13% (based on an initial tube diameter of 0.2302 as measured on tubes irradiated at zero stress). The majority of the data used in the development of the irradiation creep equation were obtained for irradiation temperatures below 450°C. Strains at 550°C, which are higher than predicted from the low-temperature data, could be a result of either a thermal creep contribution or to a temperature dependence of irradiation creep. E. R. Gilbert and J. L. Straalsund, in a HEDL study, have measured the thermal creep of 20% cold-worked Type 316 stainless steel pressurized tubes at 565°C and obtained strains of 0.041, 0.088, and 0.19% at stresses of

10,000, 20,000, and 30,000 psi, respectively, in tests lasting 4100 hr. In this experiment the tubes irradiated at 550°C were at temperature for about 3450 hr. If these thermal creep strains are simply added to the calculated irradiation creep strains, the observed strain still cannot be accounted for (i.e., at 30,000 psi the expected strain would be only 0.44% compared to the observed value of 0.74%). Since no swelling is expected in cold-worked material at this fluence, these results suggest that low-fluence irradiation creep may be temperature dependent. This is consistent with the data, in a study by E. R. Gilbert and J. L. Straalsund of HEDL, for pressurized tubes irradiated in the range 377-577°C.

TABLE ORNL.3
RESULTS OF DIAMETER MEASUREMENTS ON TYPE 316 STAINLESS STEEL TUBES^a
IRRADIATED AT 450°C in SUBASSEMBLY X100A

Condition	Estimated Fluence >0.1 MeV (n/cm ²)	Hoop Stress (psi)	Diameter (in.)	Diameter Change ^b (%)
Annealed	2.9 x 10 ²²	0	0.2299	-0.04
Annealed	2.9	7,500	0.2301	0.04
Annealed	2.9	20,000	0.2302	0.09
20% Cold- Worked	2.3	0	0.2303	0.13
20% Cold- Worked	2.3	7,500	0.2302	0.09
20% Cold- Worked	2.3	20,000	0.2305	0.22
20% Cold- Worked	2.9	0	0.2302	0.09
20% Cold- Worked	2.9	7,500	0.2301	0.04
20% Cold Worked	2.9	20,000	0.2305	0.22

^aTubes are from Carpenter Steel Heat V87210.

^bUsing 0.2300 as initial diameter.

The four holders for investigating the in-reactor fracture behavior of preirradiated Type 304 stainless steel have been disassembled. Visual examination, length, and density measurements, on holder bodies and individual samples have been completed. A portion of the results are summarized in Table ORNL.4. This table lists the neutron fluence and irradiation temperature during the present experiment as well as the fluence and temperature during the previous irradiation of the material as an EBR-II thimble.

TABLE ORNL.4
RESULTS OF IN-REACTOR SWELLING DRIVEN FRACTURE EXPERIMENT
ON PREIRRADIATED TYPE 304 STAINLESS STEEL

Holder	Neutron Fluence >0.1 MeV (n/cm ²)	Irrad. Temp. (°C)	Sample	Previous Fluence >0.1 MeV (n/cm ²)	Previous Irrad. Temp. (°C)	Previous Density Change (%)	Present Experiment		
							Length Change (in.)	Density Decrease (%)	Strain ^a (%)
X-6	1.3x10 ²²	400	Y-3-A	8.7x10 ²²	418	7.08	0.0100	2.28	-0.7
			Y-5-A	7.2	427	5.11	0.0157	2.17	0.3
			X-4-B	8.8	387	3.83	0.0087	1.96	-0.6
			Cont-2	0		0	0.0041	0.76	-0.1
Y-6	1.3	450	Y-14-B	2.1	465	0.30	0.0179	1.25	1.7
			B-21-B	1.1	370	0.24	0.0142	0.74	1.5
			X-6-A	7.5	377	2.98	0.0176	2.16	0.66
			Cont-8	0		0	0.0103	0.76	1.3
X-5	1.75	550	X-4-A	8.8	387	4.28	0.0296	2.74	2.5
			Y-14-A	2.1	465	0.30			Fracture ^b
			B-21-A	1.1	370	0.24		1.74	Fracture ^c
			Cont-1	0		0	0.0193	0.96	2.3
Y-5	1.75	550	X-6-B	7.5	377	2.98	0.0338	3.06	3.0
			Y-3-B	8.7	418	7.08	0.0319	3.52	2.3
			Y-5-B	7.2	427	5.11	0.0339	3.79	2.4
			Cont-5	0		0			

^a $\frac{(\Delta l)_{\text{Measured}} - (\Delta l)_{\text{Calculated}}}{0.625} = \text{Strain } (\%)$ where (Δl) calculated is length change calculated from density change.

^bFracture occurred in shoulder.

^cFracture strain is calculated to be less than 3.7%.

Two samples, both contained in holder X5, irradiated at 550°C to 1.75 x 10²² n/cm² (>0.1 MeV) were fractured. One sample, Y-14-A, was fractured in the shoulder with the fracture originating at the pin holes as shown in Figure ORNL 9(a). The other sample, B-21-A, fractured in the gage section as shown in Figure ORNL.9(b). In each case the fractures were predominately intergranular. The entire fracture surface of sample B-21-A is shown in Figure ORNL-10. At higher magnification, oxide was clearly visible on the grain boundary fracture surfaces (Figure ORNL.11), indicating that the fracture occurred while the sample was at temperature (i.e., during irradiation).

The strain, in excess of that attributable to the density decrease, can be calculated from length change and density change data, and is given in Table ORNL.4. Ideally, the length change for the four samples contained in any one holder should be equal. As can be seen in Tables ORNL.1, .2 and .3, this is not the case. The different length change values for different samples from the same holder are probably a result of deformation of the pin and of the sample near the pin and of errors in length measurements. The strain was calculated from the difference in the measured length change and that calculated from the density decrease of the sample with the assumption that the strain occurred over the 0.625-in.-gage section. Negative strains were calculated when the length change was less than that expected from one-third the density decrease. This occurred when the swelling rate of the sample was greater than that of the holder (i.e.,

FIGURE ORNL.9. Fracture of (a) Sample Y-14-A that Occurred at the Pin Hole (Approximately 15X). (b) Sample B-21-A that Occurred in Gage Section (Approximately 3x). (Scanning electron microscope examination showed the fracture to be intergranular.)

FIGURE ORNL.10. Fracture of Specimen B-21-A. [Type 304 stainless steel irradiated at 370°C to 1.1×10^{22} n/cm² (>0.1 MeV), then irradiated in stress effects experiment at 550°C to 1.75×10^{22} n/cm² (>0.1 MeV). (40X)]

FIGURE ORNL.11. Oxide on Grain Boundary Fracture Surface of Specimen B-21-A from Stress Effects Experiment. [Presence of oxide indicates sample fractured at temperature during irradiation. (1000X)]

for the samples irradiated at 400°C). At higher irradiation temperatures, the high-purity Type 316 stainless steel holder swelled at a faster rate than the samples and positive strains were measured.

The fracture strains measured in postirradiation creep-rupture tests⁽³⁾ are on the order of 0.1%. The strains that occurred were greater. From density measurements on sample holder X5 and on the fractured sample B-21-A it is calculated that the fracture strain for this sample was less than 3.7%. Other samples had strained by as much as 3.0% without failing.

Estimates of the stress levels that existed in the samples can be obtained from correlations between irradiation induced creep strain, stress, and swelling.⁽⁴⁾ The creep strain, ϵ , is given by

$$\epsilon = \sigma \{ A [1 - \exp(-f/B)] + cf + DS \}$$

where

σ = stress (psi)

f = fluence, n/cm^2 , >0.1 MeV

$S = S_o (1 + P\sigma_h) = \text{fractional swelling}$

S_o = stress free swelling

$P = 3.75 \times 10^{-3} S_o, S_o < 0.04$

$15 \times 10^{-5} S_o, S_o > 0.04.$

For samples X-6-B from holder Y-5, a tensile stress of 50,000 psi would have been required to produce the observed 3.0% plastic strain (assuming that a stress of this magnitude existed during the entire irradiation). However, the rupture life of the thimble material irradiated to the fluence level of $7.5 \times 10^{22} \text{ n/cm}^2$ (>0.1 MeV) is less than 1 hr at 550°C and a stress of 35,000 psi. It thus seems likely that the stress during irradiation was below this value. This in turn suggests that irradiation creep rates in materials that are swelling are higher than calculated from the correlation given in Equation (1). The 20% cold-worked Type 316 Stainless Steel pressurized tubes contained in this same experiment, also at a temperature of 550°C, exhibited 0.74% diameter change at $2.4 \times 10^{22} \text{ n/cm}^2$ at 30,000 psi. Adjusting for the difference in fluence by assuming a linear fluence dependence, the strains expected at $1.75 \times 10^{22} \text{ n/cm}^2$ (>0.1 MeV) and 30,000 psi would be about 0.5%. It thus seems likely that the creep rate is dependent on the swelling rate and possibly more strongly than suggested by Equation (1).

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II. CONTROLLED THERMONUCLEAR REACTOR PROGRAM

A. EFFECTS OF HIGH-HELIUM-GENERATION RATES ON RADIATION DAMAGE IN STAINLESS STEELS

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Purpose

These investigations are directed at determining 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 CTRs.⁽¹⁾ The same suggestion was forwarded by P. Bonanos and W. Price, Jr., of the Plasma Physics Laboratory, Princeton, New Jersey. At a neutronic wall loading of 1.87 MW/m^2 , approximately 30 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.⁽²⁾ In alloys containing nickel, the helium concentration has been found experimentally to increase as $(\phi t)^{1.68}$ (Reference 4), 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.

Previously Reported Results

Annealed and 20% cold-worked specimens of standard Type 316 stainless steel were irradiated in HFIR at temperatures between 370 and 785°C to fluences $\leq 8.7 \times 10^{22} \text{ n/m}^2$ ($>0.1 \text{ MeV}$). Details of specimen chemistry and processing and of the irradiation experimental methods and irradiation parameters were reported previously.^(4,5)

Results of swelling determined by immersion density measurement and transmission electron microscopy on one sample, presented previously,^(4,5) 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 that predicted by displacement damage (EBR-II results) and is somewhat less than that 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 cold-worked samples and increases slightly in annealed and rapidly in cold-worked samples at 680°C.

Postirradiation tensile and creep-rupture test results were also reported previously.⁽⁶⁾ Samples irradiated in the annealed condition exhibited an increase in yield strength at irradiation and test temperatures between 350 and 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% cold-worked 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. Postirradiation creep-rupture tests of both the annealed and cold-worked structures exhibited large reductions in rupture life and ductility due to irradiation.

Optical metallography of these samples⁽⁷⁾ showed that extensive precipitation had occurred for all irradiation conditions studied. Intragranular precipitation in temperature-time regimes where thermal aging showed no precipitation thus indicates an enhancement of the precipitation kinetics produced by the irradiation. Although other precipitate phases are possible, it appears that the precipitate was mainly $M_{23}C_6$ at lower temperatures and a mixture of $M_{23}C_6$ and sigma phase at higher temperatures (685°C). Sigma phase precipitation occurred mainly on grain boundaries. $M_{23}C_6$ precipitated intragranularly and in the cold-worked samples precipitated on deformation bands. A more detailed description and representative micrographs were included in the previous progress report.⁽⁷⁾

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, showed the lower temperature fracture to be predominately transgranular. The sample exhibited very little necking or reduction of area and only the very outer rim appeared to fail in shear. At 650°C, the fracture was clearly intergranular.

Sample Preparation for Electron Microscopy

The high specific radioactivity of these samples irradiated in the mixed spectrum of HFIR prevented the preparation of thin section foils for electron microscopy by conventional laboratory techniques. As a result, techniques were developed for fully remote sample preparation in the hot cells. The specimens to be thinned were sectioned from the gage section of the mechanical properties samples. The resulting samples were 2 mm in diameter and averaged approximately 0.15 in. (0.4 mm) thickness. The activity of these samples before thinning was greater than 10 R/h at contact. This radiation is very penetrating and difficult to shield.

The ORNL "standard" two-step semiautomatic technique⁽⁸⁾ was successfully adapted for use in the hot cells. This technique involves first dimpling the specimen on each side in turn with an electrolytic jet and then polishing uniformly in a standard electrolytic cell. Polishing is continued until penetration occurs. A light beam and photo cell detector is used to halt polishing as soon as a hole forms. The jet dimpling proved to be the critical step for hot cell use of this technique. The difficult alignment of jet and specimen was accomplished by use of both periscope and through-window telescope viewing. The jet was reduced to minimum size by use of a 0.3 in. (0.8 mm) diameter nozzle. The main other requirements were experience and patience on the part of the operators. The electrochemical conditions used to prepare successful specimens were:

Jet Dimpling: Solution of 8% perchloric acid, 10% butyl cellosolve, and 12% water in ethyl alcohol.

Current of 150 ma with 0.3 in. (0.8 mm) nozzle.

Temperature 33 to 35°C.

Electropolishing: Solution of 6% perchloric acid in ethyl alcohol.

Current of 0.3 amp on 0.08-in. (2-mm)-diameter sample with stainless steel cathodes.

Temperature ~5°C.

Microstructure of Irradiated Samples

Samples for TEM analysis were cut from the center of the gage length of the tested mechanical properties specimens. Specimens were cut well back from the fracture and, because of the near zero values of uniform elongation, there is little likelihood of the microstructure being affected by the tensile tests. Thin-section samples were prepared as described in the previous section and these were examined in a Hitachi HU 200E electron microscope operating at 200 kV. Examination of microstructures included thickness determinations on stereo pairs of photographs and size classification of voids on a Zeiss Particle Size Analyzer.

Table ORNL.5 gives the irradiation conditions for specimens that are included in our initial microstructural examination matrix. These are samples on which swelling and mechanical properties have been reported earlier.⁽⁴⁻⁷⁾

TABLE ORNL.5

IRRADIATION CONDITIONS AND IMMERSION DENSITY CHANGES FOR TYPE 316 STAINLESS STEEL ELECTRON MICROSCOPY SAMPLES

Sample	Irradiation Temperature (°C)	Fluence >0.1 Mev (n/m ²)	Displacements ^a (dpa)	Helium ^b (ppm)	Swelling ^c from Density (%)
<u>Solution Annealed</u>					
D0-5	528	8.3 x 10 ²⁶	114	5450 [*]	8.33
D0-2	546	6.2	85	3190	NA ^d
D0-7	679	8.7	121	6090	14.1
<u>20% Cold-Worked</u>					
D2-5	379	7.1	97	4020	1.59
D2-6	456	7.7	107	4820	0.80
D2-7	528	8.3	114	5450	2.55
D2-8	602	8.7	119	5940	3.29
D2-9	679	8.7	121	6090	16.8

^aThe dpa calculated from Lindhard model. H. T. Kerr and R. Q. Wright, Oak Ridge National Laboratory, personal communication (May 17, 1973).

^bAtomic concentration calculated by the formula $C = 9.8 K \times 10^{-19} (\phi t)^{1.68}$ atoms He/g Ni, where ϕt is thermal neutron fluence. Formula from McElroy and Farrar in "Radiation-Induced Voids in Metals," J. W. Corbett and L. C. Ianniello, Eds., AEC Symp. Ser. 26, p. 202, CONF-710601 (April 1972), with normalization factor K = 0.65 determined from measured values.

^cF. W. Wiffen and E. E. Bloom, Nucl. Technol. 25: 113 (1975).

^dNA = not available.

The preliminary examination of samples D0-2 and -7 of the annealed material and D2-5, -6, -9 of the cold-worked material has been completed. The following facets of the microstructure will be examined and compared among the samples: cavity morphology, preferential cavity locations, dislocation structure, precipitate size, distribution, and shape, and grain-boundary features.

Looking first at the 20% cold-worked samples, the lower temperature (379 and 456°C), samples had a very high concentration of small round or polyhedral shaped cavities. [Figures ORNL.12(a) and (b).] The high-temperature sample (679°C), had a much lower concentration of very large cavities having a square or rectangular projection. Since extensive data as to average cavity dimensions and cavity concentrations have not been compiled, the comparisons made between samples are only relative. Most of the data are qualitative, with quantitative data to follow in subsequent reports. More meaning can be given to the comparisons by referring to Figures ORNL.12, .13 and .14. The cold-worked sample irradiated at 379°C contained a few cavities that had much larger diameters than the average. These large cavities seem to be preferentially attached to blocky precipitates in the deformation bands of the cold-worked material. The 456°C microstructure does not show the large cavities exhibited by the 379°C sample, and in addition, the precipitate structure and the cavity structure are different in the 456°C sample than in the 379°C sample. The sample irradiated at 456°C has long thin precipitates arranged in a patch-work or checker-board fashion and what seems to be a lower cavity concentration also. This is consistent with the smaller observed density decrease for the 456°C sample than for the 379°C sample. It is possible that the precipitate structure has suppressed cavity formation even though the 456°C sample was irradiated to a higher displacement level and a higher helium concentration. The cavities in the cold-worked sample irradiated at 679°C are larger and of lower concentration than at the lower temperatures [Figure ORNL.12(c)]. All show a regular polyhedral shape, but the crystallography of bounding planes has not yet been determined.

Both annealed samples examined exhibit a high concentration of large cavities [Figures ORNL.13(a) and (b).] In the 546°C sample, as in the 456°C cold-worked sample, there are two scales of damage. There are smaller more uniformly distributed cavities throughout the matrix, while there are a few very large cavities attached to two or three long precipitate particles. The difference between the average size and the largest size is not as marked as in the 679°C sample. The cavities in the 679°C sample project as squares or rectangles, and are assumed to have the same crystallography as the cold-worked sample irradiated at the same temperature. The cavities in the 548°C sample are rounded or polyhedral shaped, with somewhat less regular crystallography than the cavities in the 679°C sample.

In both the cold-worked and annealed samples, it seems that the very large cavities are preferentially attached to one or several precipitates. In both the cold-worked and annealed samples at 679°C, there is a higher concentration of smaller than average and average sized cavities on the interface between precipitates and matrix. In the annealed samples at 546°C, there is a larger concentration of small cavities near and on the interface between precipitates and matrix. On a much finer scale, the 456°C sample of the cold-worked series also shows cavities of about the same size as the average size attached to almost all precipitate-matrix interfaces. The cavity concentration at the interface seems about the same as the cavity concentration in the matrix. There are far fewer precipitates in the 379°C cold-worked

FIGURE ORNL.12. Microstructures of 20% Cold-Worked Type 316 Stainless Steel After Irradiation in the HFIR. (a) Irradiated to $7.1 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$) at 379°C , Containing 4020 ppm He. (b) Irradiated to $7.7 \times 10^{26} \text{ n/m}^2$ at 456°C , Containing 4820 ppm He. (c) Irradiated to $8.7 \times 10^{26} \text{ n/m}^2$ at 679°C , Containing 6090 ppm He.

FIGURE ORNL.13. Microstructures of Annealed Type 316 Stainless Steel After Irradiation in the HFIR. (a) Irradiated to $6.2 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$) at 546°C , Containing 3190 ppm HE. (b) Irradiated to $8.7 \times 10^{26} \text{ n/m}^2$ at 679°C , Containing 6090 ppm He.

FIGURE ORNL.14. Photomicrographs Containing Grain Boundaries from a Few of the Irradiated Type 316 Stainless Steel Samples. (a) Grain boundary from annealed sample irradiated at 546°C showing large, blocky, precipitates along the grain boundary and dislocation segments in a portion of the grain. (b) Grain boundary from annealed sample irradiated at 679°C showing cavities oriented along different relative directions in each grain. (c) Grain boundary from cold-worked sample irradiated at 679°C showing the grain boundary in diffracting condition and dislocation segments connecting the cavities.

sample and few if any of the large rod shaped precipitates found in the 456°C cold-worked and 546°C annealed samples. There is also little or no observed cavity-precipitate interface interaction in this sample.

We have not yet examined grain boundaries in the 379 and 456°C cold-worked samples. Grain boundaries in the 546 and 679°C annealed samples and in the 679°C cold-worked samples are shown in Figure ORNL.14. The grain boundaries observed in the 546°C annealed sample are lined with chunky precipitates and have a large concentration of very small cavities near and attached to the precipitate-matrix interface, but there is a denuded region near the grain boundary that does not contain the average size cavities found in the matrix. In the 679°C cold-worked sample and the 679°C annealed sample the distribution of cavities seems to be unaffected by the grain boundaries.

The dislocation structure is difficult to observe in the 379 and 456°C cold-worked samples, however, it looks like patches of tangled segments uniformly distributed throughout the sample. The dislocation structure in the 546 and 679°C annealed samples and the 679°C cold-worked sample consists of line segments pinned at cavities and precipitates. There is no observed case of a dislocation segment not being attached at one end to a cavity or precipitate. No faulted or unfaulted loops were observed in any of the samples.

The precipitate size and distribution vary from sample to sample. In the 379°C cold-worked sample, there are rather small, blocky precipitates in close conjunction with the slip bands. In the 456°C cold-worked sample, there is a high density of the smaller blocky precipitates as in the previous 379°C sample, and they are much more uniformly distributed throughout the matrix. The distribution of precipitates in this sample is associated with the finer slip band in the areas examined. In addition, there is also a high density of rod-like precipitates that are about four to five times larger than the smaller precipitates, and they are distributed uniformly along the slip bands. In the 679°C cold-worked sample, there are only a few very large blocky precipitates that seem randomly distributed throughout the matrix. In the 546°C annealed sample, there are large, blocky precipitates along the grain boundaries, as mentioned previously, and a combination of huge rod-like and blocky precipitates distributed randomly throughout the matrix. The 679°C annealed sample exhibits the same behavior as the 679°C cold-worked sample, namely a few very large blocky precipitates distributed randomly through the matrix.

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EFFECT OF HELIUM ON VOID NUCLEATION ON ION BOMBARDED VANADIUM

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I. INTRODUCTION

Vanadium is one of a number of possible materials for the first wall in future thermonuclear reactors. It is also known that the wall will be exposed to intense neutron damage and internal gas buildup. Heavy ion damage in conjunction with helium preinjection is currently being used to simulate the type of damage expected in a first wall of a fusion reactor.

In the experiments to be described void microstructures were studied in vanadium which had both been preinjected with helium and with no helium, and subsequently bombarded with 46 meV Ni⁶⁺ ions. In addition, the void microstructure produced by 7.5 meV Ta⁺⁺⁺ ions in material with no helium was also studied. The void parameters were studied at several ion dose levels in the 46 meV Ni irradiation and at three temperatures in the 7.5 meV Ta irradiations. In essence, the two experiments are distinguishable as follows:

- (1) 46 meV Ni ion - low dose rate, with and without preinjected He, 750°C
- (2) 7.5 meV Ta ion - high dose rate, no He, 700-900°C

II. EXPERIMENTAL PROCEDURES

The commercially pure vanadium was in the form of foil approximately 0.15 mm thick. All material was annealed at 1100°C at 10⁻⁷ torr prior to bombardment. The irradiation conditions are described separately for the two bombardment experiments.

A. 46 meV Ni⁶⁺ IRRADIATION

The specimen, in the form of a 2 cm disc, was masked off so that only two square areas were bombarded with the beam as shown in Figure PNL.1. One of the square areas had been preinjected with 3 ppm He and the other had no helium. The 46 meV Ni⁶⁺ beam covered both areas. The temperature of the irradiation was 750°C and the vacuum was 2 x 10⁻⁷ torr. The total ion dose was 3 x 10¹⁶ ions/cm² and the bombardment lasted 5.1 hours. The irradiation was done in the Variable Energy Cyclotron at Harwell, England.

B. 7.5 meV Ta⁺⁺⁺ IRRADIATION

The specimens, which contained no helium, were 3mm discs and were covered with a face plate with a 2mm hole. The pressure of the face plate provided thermal contact to the furnace block. Specimens were bombarded with a 7.5 meV Ta⁺⁺⁺ ion beam, 3mm in diam, at 700°C, 800°C and 900°C in a vacuum of 2 x 10⁻⁶ torr. The total ion dose was 7.5 x 10¹⁵ ions/cm² and the bombardments lasted about 0.5 hours.

FIGURE PNL.1. Schematic Representation of Specimen Configuration for Bombardment with 46 meV Ni⁶⁺ Beam. [The Ni ion beam covered both squares as indicated by the dotted line. Approximately six samples were obtained from each square. (Not drawn to scale.)]

C. DAMAGE CALCULATIONS

The displacement damage produced by the ions was computed using the energy deposition program EDEP-1 of Manning and Mueller⁽¹⁾. The damage in terms of dpa was computed from the following expression:

$$\text{dpa} = \frac{0.8 \cdot \text{SD}(x) \cdot N_I}{2E_D}$$

dpa = atom displacements/atom

SD(x) = energy transfer from EDEP-1

E_D = effective threshold energy

N_I = ion dose

0.8 = correction factor

For vanadium, E_D is assumed to be 40 eV. The displacements per atom (dpa) are shown as a function of distance from the surface in Figure PNL.2 for both the 46 meV Ni⁶⁺ and 7.5 meV Ta⁺⁺⁺ ions. The arrows indicate the places at which foils were extracted for observation in the electron microscope. In addition one foil from a region very near the bombarded surface in the 46 meV Ni irradiation was also studied. The amount of surface removed from each specimen was determined by monitoring the absorption of an x-ray beam after passing through the specimen in a manner described previously⁽²⁾.

III. RESULTS AND DISCUSSION

A. 46 meV Ni⁶⁺ IRRADIATION

Figure PNL.3 shows the microstructure of a helium containing specimen from a region 4.9μ from the surface (9 dpa). Well defined voids are observed. However, there is also a very high dislocation density which is arranged in a cell structure typical of a worked material. This cell structure is not indicative of ion induced dislocation damage. The origin of this cell structure is not known but it was formed prior to irradiation.

FIGURE PNL.2. Displacement Damage vs. Penetration for 7.5 meV Ta⁺⁺⁺ and 46 meV Ni⁶⁺ Bombarded onto Vanadium. (Vertical arrows indicate region from which TEM specimens were extracted.)

FIGURE PNL.3. Microstructure of Vanadium Containing 10 ppm He After Bombardment with 46 meV Ni Ions at 750°C. (Dose level $\approx$ 9 dpa.)

Figure PNL.4 is a micrograph from another area of the same specimen shown in Figure PNL.3. There are small recrystallized grains with some unrecrystallized areas to the side. Only two such areas were observed in this specimen. In order to recrystallize during bombardment, the temperature of 750°C is close to, but somewhat less than, the normal recrystallization temperature since only a few areas recrystallized. There are no voids in this region as the grain boundary migration has swept them all up.

The microstructure from a region which had been at temperature in the accelerator but not exposed to the beam is shown in Figure PNL.5. This specimen was taken from outside the bombarded region shown in Figure PNL.1. A high dislocation density is observed but the cell structure is less obvious.

Figure PNL.6 illustrates the microstructure in ion bombarded material which had no helium preinjection. At no place within the bombarded region could voids be observed. The dislocation cell structure is present although the cell size is somewhat larger than in the helium bearing specimen. The larger cell size indicates that there was more recovery in the material which contained no helium.

The void parameters in the ion bombarded specimen containing helium are listed in Table PNL.1. The dose values are taken from the calculated curve shown in Figure PNL.2. The dose rate is relatively low and varies by a factor of 8 from the lowest to the highest value. In specimens from a region $\geq 0.5\mu$ from the surface, no voids were observed. The large voids observed at the low dose seem anomalous. However, large voids have been observed near the edge of grain boundary denuded zones. Therefore, the large voids observed here may be near the edge of the surface denuded zone.

TABLE PNL.1
VOID PARAMETERS IN ION BOMBARDED VANADIUM 46 meV Ni^{6+} IONS AT 750°C

DOSE (dpa)	DOSE RATE (dpa/sec)	VOID SIZE (Å)	DENSITY (cm^{-3})	VOID FRACTION (%)
2.4	1.2×10^{-4}	640	1×10^{13}	0.27
9	4.5×10^{-4}	300	2×10^{14}	0.54
16	8×10^{-4}	350	1.7×10^{14}	0.73

These results show that helium definitely acts as a void nucleating agent under these particular bombardment conditions. However, the second experiment will show that helium is not absolutely necessary. In the specimens without helium, the high dislocation density provides sinks for the point defects. When this factor is combined with the relatively low dose rate (i.e. defect generation rate), a sufficient supersaturation of vacancies can not be generated to promote void nuclei that are larger than the critical size. However, the critical size for a stable void nucleus is much smaller when helium atoms can stabilize the nucleus. These results are in qualitative agreement with the nucleation theory of Wiedersich et.al.⁽³⁾ who show that for the lower dose rates and higher temperature, helium is essentially necessary for void nucleation. They have also shown that as the dislocation density is increased, more helium is necessary for void nucleation.

b. 7.5 meV Ta^{+++} IRRADIATION

FIGURE PNL.4. Microstructure of Same Foil as in Figure PNL.3 But Different Area. (The recrystallized grains shown here were only observed in two areas of the foil.)

FIGURE PNL.5. Microstructure of Vanadium Taken from Large Specimen Shown in Figure PNL.1 But from Region Not Exposed to the 46 meV Nickel Beam.

FIGURE PNL.6. Microstructure of Vanadium Containing No Helium After Bombardment with 46 meV Ni^{6+} Ions at 750°C. (Dose level $\approx$ 9 dpa.)

The void microstructure in specimens bombarded with 7.5 meV Ta⁺⁺⁺ ions at 700°C is shown in Figure PNL.7. Large, distinct voids are present. There is no dislocation cell structure. Dislocations are present but out of contrast in the micrograph. The crystallographic shape of the voids is plainly evident in the figure. The voids are basically cubes with [001] faces with some truncation on the [111] planes.

Figure PNL.8 shows the void microstructure in specimens bombarded at 800°C. The voids are much larger than in the previous micrograph. The dislocations are in contrast and appear to thread the voids. Only individual dislocations and not a cell structure are observed. At 900°C, no voids are observed at all.

The void parameters for this bombardment are listed in Table PNL.2. The dose rate is much higher than in the 46 meV Ni⁶⁺ irradiation, although the total dose is comparable to the

TABLE PNL.2

VOID PARAMETERS IN ION BOMBARDED VANADIUM

TEMPERATURE	DOSE (dpa)	VOID SIZE (Å)	DENSITY (cm ⁻³)	VOID FRACTION (%)
700°C	15	330	2.5x10 ¹⁴	0.6
800°C	16	550-900	0.5-5x10 ¹³	0.7
900°C	15	No Voids	---	---

highest dose in the nickel irradiation. The swelling values are also comparable. The void density is very inhomogeneous at 800°C and consequently the swelling values may not be too accurate. The absence of voids at 900°C is either a result of irradiating beyond the temperature limit of void formation or being too close to the surface for this particular temperature.

The results of the Ta ion irradiation show that voids can be produced in annealed vanadium without the presence of helium. The higher dose rate and lower dislocation density result in a higher net defect generation rate and hence promote homogeneous void nucleation. This also is in at least qualitative accord with the nucleation theory of Wiedersich, et.al.⁽³⁾, as discussed previously.

The only similar study on ion bombarded vanadium has been done by Santhanam, et.al., who bombarded vanadium containing both 10 and 100 ppM helium⁽⁴⁾. It is difficult to compare the results directly since Santhanam, et.al., had different purity material, bombarded to a higher dose and did not bombard specimens containing no helium. However, it appears that the void sizes were smaller in that work and the void density higher when the difference in dose levels is taken into account. This could be a result of the greater amount of helium in their specimens.

Summary

These experiments illustrate that under certain material and irradiation conditions, helium is a necessary ingredient for void nucleation. In the vanadium with a high dislocation density, helium is necessary for void nucleation at least when bombarding at dose rates of 10⁻⁴ to 10⁻³ dpa/sec at 750°C. In the annealed vanadium, no helium was required for void nucleation at least in the temperature range 700-800°C when the dose rate was as high as 10⁻² dpa/sec. These results are in qualitative agreement with current void nucleation theories.

Figure PNL.7. Microstructure of Vanadium Containing No Helium After Bombardment with 7.5 meV Ta⁺⁺⁺ Ions at 700°C. (Dose level $\sim$ 15 dpa.)

FIGURE PNL.8. Microstructure of Vanadium Containing No Helium After Bombardment with 7.5 meV Ta^{+++} Ions at 800°C. (Dose level $\approx$ 16 dpa.)

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University Of Cincinnati

RADIATION EFFECTS ON THE BCC METALS AND ALLOYS

John Moteff

I. OBJECTIVE

The objective of this program is to evaluate the influence of several BCC metals and alloys with the ultimate view that the substructure will influence the elevated temperature mechanical properties. The variables studied include: irradiation temperature (430, 580, 700, 800, 900 and 1050°C); base metal (Mo, Nb and W); dilute alloys (Mo-0.5 Ti, Nb-1Zr); high concentration alloy (W-25 Re); thermal stability of the defect state based on postirradiation annealing temperatures.

In particular, the nature and the number density of observable irradiation induced defect clusters are determined by the use of a 200 kV transmission electron microscope (TEM). In addition, the nature and number density of defect clusters as a function of annealing temperature is also investigated principally by the use of the TEM. The TEM observations are supplemented by other property measurements such as bulk density, electrical resistivity and dimensional change measurements.

Finally, the influence of the irradiation induced defect clusters on the mechanical properties (i.e. dislocation interactions with irradiation induced defects) of these metals and alloys are evaluated, initially by means of microhardness measurements to be followed by tensile and creep tests on miniaturized specimens.

II. INTRODUCTION

The work performed during this period consisted of (a) a completion of the determination of the bulk density changes of Mo, Mo-0.5 Ti, Nb, Nb-1Zr, W and W-25Re in the as-irradiated ($\sim 1 \times 10^{22}$ n/cm², $E_n > 1$ MeV in EBR-II, Row 7 at 430, 580, 700, 800, 900 and 1050°C) condition, (b) additional data reduction of the detailed TEM investigations of Mo and Mo-0.5 Ti in the as-irradiated and the annealed condition, (c) initiation of an ion (Vanadium) bombardment study on Mo, Mo-TZM and Mo-0.5 Ti through a Thesis Parts Program with Argonne National Laboratory, at different dpa levels, which includes the dpa level of the EBR-II irradiated Mo and Mo-0.5 Ti samples, and at temperatures ranging from about 800 to greater than approximately 1450°C, (d) detailed TEM study of the Nb specimens in the as-irradiated and the annealed (~ 430 up to $\sim 1300^\circ\text{C}$) condition and the (f) data reduction on the resistivity measurements of the W and W-25 Re specimens in the as-irradiated and the annealed condition.

In cooperation with other AEC (ERDA) sponsored programs, the University of Cincinnati provided (a) several molybdenum specimens containing the void superlattice to Dr. Seidman at Cornell University for Field Ion Microscopy (FIM) studies and (b) six Nb and six Nb-1Zr specimens, representing one of each irradiation temperature to Dr. Carl Koch at the Oak Ridge National Laboratory for use in his superconductivity studies. Other areas of interaction in the effects of irradiation on BCC metals is with Dr. J. L. Brimhall of the Pacific Northwest Laboratories in their program on the calibration studies of ion irradiated molybdenum.

III. SWELLING BASED ON IMMERSION DENSITY MEASUREMENTS

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Preliminary evaluation of bulk density changes in the six different metals as a function of the neutron irradiation temperature is given in Figure UC.1. The swelling for the pure metals ranged from approximately 0.2% up to 1.9% and suggests a definite temperature dependence.

It is quite apparent that the alloy condition influences the swelling in the three base metals in different ways. For instance, adding 1% Zr to the Nb resulted in limiting the void swelling to only the irradiated temperature of 800°C, although swelling was observed in Nb for all six temperatures. The addition of 0.5% Ti to Mo progressively increased the swelling tendency from about 20 to 50% over that of Mo at the five higher temperatures.

For the case of W-25 Re, which is normally a solid solution alloy, it is shown that the irradiation induced swelling was essentially zero within the experimental error with the exception of a slight increase to occur at the higher temperature. TEM observations performed on this alloy indicate that a large fraction of a second phase has precipitated.⁽¹⁾ This phase is relatively stable up to temperatures in the region of 1550 to 1700°C. No voids or bubbles have been observed in this alloy either in the as-irradiated or the completely annealed condition. Dimensional measurements⁽²⁾ made on the irradiated tungsten during the course of the resistivity annealing studies suggest that the macroscopic swelling is strongly anisotropic and that rolling and/or drawing texture of the specimens may be the reason even though they have been given a recrystallization heat treatment.

As clearly shown in the plot of the swelling as a function of the homologous temperature (Figure UC.2), the observed swelling of the three unalloyed metals tends to occur in two different temperature regions lying between 0.19 and 0.35 T_m and between 0.35 and 0.50 T_m . This tendency for a double peak to occur in the swelling of BCC metals is not understood at the present time. The results are in qualitative agreement with studies on Nb by Loomis, Taylor and Gerber of ANL.⁽³⁾

Table UC.1 lists several normalized parameters for Nb, Mo, and W which may be considered important in the evaluation of irradiation induced defect behavior. Particularly, the relative values of the self-diffusion activation energies and the shear moduli of the three elements. This circumstance suggests that diffusion controlled behavior of Mo and Nb may be quite similar and that of tungsten being approximately 40% greater, sufficiently different for measurable observations in any diffusion controlled behavior in the temperature range of this experiment. On the other hand, the modulus related parameter suggests that W and Mo are within about 12% of their average, both being factors of three to four times greater than that of Nb. This argument, of course, will be strongly dependent on the influence which the impurity level in the metals, especially in the case of Nb, will have on these properties. The number of displaced atoms per neutron for the case of Nb and Mo should be roughly twice that of W in this experiment.

FIGURE UC.1. Comparison of the Swelling Characteristics of Refractory Metals and Alloys as a Function of Neutron Irradiation Temperature (Fluence = 1×10^{22} n/cm², $E_n > 1$ MeV).

FIGURE UC.2. Comparison of the Swelling Characteristics of Nb, W and Mo on the Basis of the Homologous Temperature (Fluence = 1×10^{22} n/cm², $E_n > 1$ MeV).

TABLE UC.1

SOME RELEVANT PROPERTIES OF THE BCC METALS

Property	Approximate Normalized Values		
	Nb	Mo	W
Diffusion Energy	1.0	1.1	1.4
Shear Modulus	1.0	3.6	4.5
Atomic Weight	1.0	1.0	2.0
Cross Section*	1.0	1.0	1.0

* 1 MeV neutron

In view of the fact that both the tungsten and Mo show similar swelling behavior based on a homologous temperature plot clearly indicates that the diffusion controlled parameters play an important role in void swelling, unless there is a compensating effect of the number of atom displacements and/or that a saturation steady state has been reached in both metals for these irradiation conditions.

Assuming that the impurity level in Nb will not increase the shear modulus to values approaching that of Mo or affect the self-diffusion substantially, then the difference in the swelling in a region of the first peak may be attributed to the difference in the shear modulus between the Mo and Nb specimens with the swelling tendency decreasing with a decrease in the modulus.

IV. ION IRRADIATED MOLYBDENUM

J. Stubbins

At the present time one graduate student (J. Stubbins) is in the process of completing his thesis research on vanadium ion irradiated molybdenum through a Thesis Parts Program with Argonne National Laboratory. This experimental program performed with the cooperation of Dr. A. Taylor of ANL, uses the Dynamatron Facility and is designed to study the effect of ion irradiation temperature on the void characteristics of Mo, Mo-TZM and Mo-0.5 Ti. A fixed atom displacement, similar to that of the neutron irradiations described above, is considered for the purpose of direct comparison. A fluence dependency at several irradiation temperatures was also investigated. Preliminary comparisons of the calculated swelling behavior of the ion irradiated molybdenum, based on TEM void measurements are shown in Figure UC.3. The corresponding TEM determined swelling of the neutron irradiated materials shows a much lower swelling behavior. It should be pointed out that a temperature shift to correct to an effective temperature for the ion irradiated data was not used in the plot of Figure UC.3.

V. ORDERED VOID LATTICE

Experimental data on the ordered void lattice, observed to form in molybdenum⁽⁴⁾ and other metals following irradiations in high flux density reactors and/or ion bombardment facilities, as well as many proposed theories and explanations, have been reported extensively in the literature. In reviewing the nature of the data in this area, it is quite apparent that the information is rather inconclusive, in that different irradiation temperatures, atom displacement rates, bombarding ions, materials and some materials of different purity levels were used in the various experiments.

In the present investigations at the University of Cincinnati, several important observations have been made on the ordered void lattice. The significance of this data is attributed to the fact that identical material was irradiated at the same time (i.e. consistent reactor operating history and identical neutron spectrum) to six widely different temperatures. Therefore, the experimental results should contribute more meaningful information to the subject of the void superlattice.

Perhaps the most important observation is that the void lattice is strongly temperature dependent, a fact that is not now treated in any of the existing theories. For instance, in the temperature range of 430 to 700°C the lattice constant a in Angstrom units will vary linearly with the absolute irradiation temperature T in degrees Kelvin, i.e., $a = mT$ where

FIGURE UC.3. Comparison of the Swelling Characteristics of Neutron and Vanadium Ion Irradiated Molybdenum as a Function of Homologous Temperature.

m is equal to $0.32 \text{ \AA } ^\circ\text{K}^{-1}$. This unique relationship between the lattice constant and the irradiation temperature is the basis of a recommendation that this phenomena be put to use as a reactor temperature monitor⁽⁵⁾ for supplementing the silicon-carbide detectors and/or other devices that are presently being used in uninstrumented irradiation capsules.

From the preliminary studies on specimens irradiated in EBR-II at six different temperatures, the following observations are noted:

- Voids and an ordered void lattice form in molybdenum during irradiations at temperatures as low as 430°C ($0.24 T_m$) and in tungsten (6) at temperatures as low as 580°C ($0.22 T_m$).
- The ordered void lattice parameter increases linearly with the irradiation temperature for both tungsten and molybdenum in the temperature region of about 0.22 to $0.34 T_m$ and the curves of the lattice parameter versus the homologous temperature are coincident.

- c) A poorly defined ordered void structure is observed at neutron irradiation temperatures greater than 700°C. The void lattice in molybdenum is as high as 660Å for an irradiation temperature of 1050°C. A plot of the lattice parameter as a function of irradiation temperature for molybdenum is given in Figure UC.4.

FIGURE UC.4. Temperature Dependence of the Superlattice Void Parameter in Neutron Irradiated Molybdenum (Fluence = 1×10^{22} n/cm², $E_n > 1$ MeV).

- d) The voids are faceted even at the 430°C irradiation temperature and the faceting is quite similar to that reported for the case of low fluence irradiations.
- e) The void ordering at an irradiation temperature of 1050°C shows that the (100) truncating planes are stacked parallel to each other.
- f) With the exception of the 430°C irradiation temperature, the mean distance between voids in the random orientation is approximately the same as the lattice parameter of the superlattice, i.e. $a\lambda$ where $\lambda = 1/N_V^3$ with N_V being the void number density for the random void distribution.
- g) Results of the lattice parameter from ion irradiated samples has been shown⁽⁵⁾ to be in reasonable agreement with the lattice parameter of neutron irradiated specimens, provided an effective temperature T_e is used for the ion irradiated values. The correction that was used is:

$$1/T_e = (1/T_i) + (k/E_m^V) \ln (K_i/K_n)$$

where E_m^V is the vacancy migration energy, T_i is the actual ion irradiation temperature, K_i and K_n are the atom displacement rates for the ion and the reference neutron irradiations respectively.

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I. IRRADIATION DAMAGE TO REACTOR METALS

T. T. Claudson

A. LOW TEMPERATURE CREEP OF 20% COLD-WORKED TYPE 316 STAINLESS STEEL

L. D. Blackburn, J. J. Holmes

1. Objective

The objectives of this work are: (1) to accurately measure thermal creep deformation in 20% cold-worked Type 316 stainless steel, and (2) to further demonstrate the application of microwave extensometers for high precision creep strain measurements.

2. Accomplishments and Status

a. Scope of Work. A creep test on 20% cold-worked Type 316 stainless steel using a high precision microwave extensometer to measure strain was continued to further explore creep response under varying load and temperature. This test was initiated at 850°F, and the load history and strain response were reported previously.^(1,2) The temperature was then raised to 1000°F, and the specimen was stressed to 20,000 psi for 960 hours, unloaded to 220 psi 240 hours, reloaded to 20,000 psi for 625 hours, and unloaded again to 220 psi for 335 hours. After this final unloading, the test temperature was increased to 1100°F in 5 hours, and then held constant for 43 hours. The specimen was then loaded in increments of about 2,000 psi to a total stress of 20,000 psi within about 7 minutes. After 980 hours at a stress of 20,000 psi, the specimen was unloaded to 220 psi for a period of 290 hours and then reloaded to 20,000 psi.

b. Results. Strain measurements obtained during loading at 1100°F are summarized in Figure HEDL.1, along with earlier results at 850 and 1000°F. The small departure from elastic behavior at high stresses (i.e., a strain of 18×10^{-6} at 20,000 psi) is probably a result of creep during the finite loading time. A value of 21.6×10^6 psi was obtained for Young's modulus at 1100°F; this value is slightly lower (by about 4%) than the results reported by Garofalo et al.⁽³⁾

Complete results of creep strain measurements at both 1000 and 1100°F are presented in Figures HEDL.2 and .3. Since the elastic extensions and contractions accompanying stress changes were larger than the time dependent deformation, these strain contributions were subtracted out and are tabulated separately in the figures. These elastic strain measurements agree to within about one percent.

The previous report⁽²⁾ presented 1000°F results through about 260 hours following the reload. The additional results in Figure HEDL.2 confirm the observation that the measured strain after reloading is slightly larger than predicted from reoccurrence of anelastic strain and continued plastic creep strain. Recovery strains following the second unloading are very similar to those observed during the first unloading for short times, but are slightly smaller at long recovery times.

The initial creep rate is substantially higher at 1100°F than at the lower temperatures (Figure HEDL.3), but creep rate decreases more rapidly with time, so that after 1000 hours,

HEDL 7412-22.22

FIGURE HEDL.1. Loading Curves for 20% Cold-Worked Type 316 Stainless Steel at 850, 1000 and 1100°F.

HEDL-3

HEDL 7412-22.21

FIGURE HEDL.2. Creep Curve for 20% Cold-Worked Type 316 Stainless Steel at 1000°F.

HEDL 7412-22.25

FIGURE HEDL.3. Creep Curve for 20% Cold-Worked Type 316 Stainless Steel at 1100°F.

similar creep rates are observed at 1000 and 1100°F. As a result, the creep strain at 1100°F is only about 28% larger than the creep strain at 1000°F after 1000 hours. At 1100°F, strain recovery occurs rapidly after unloading and the rate of strain recovery is higher than the initial forward creep rate, as shown in the inset of Figure HEDL.3. This behavior is observed at each of the three test temperatures. Creep strain accrues rapidly after reloading, but the observed strains are slightly smaller than predicted from a reoccurrence of anelastic strain and continued plastic creep strain, as shown by the construction in Figure HEDL.3.

c. Evaluation of Results. The creep curves after the first loading show a progressive increase in creep strain with increasing temperature (see Figure HEDL.3), but the creep rate at 1100°F seems to decrease more rapidly as deformation proceeds than we would expect. That is, the creep rate after 1000 hours does not reflect a strong temperature dependence in the 1000-1100°F range. One possible explanation for this observation is that the prior creep deformation at 1000°F hardens the material so that creep rates at 1100°F are then lower than for a virgin specimen. To examine the present results for this history effect, we compared our results with those obtained from pressurized tube specimens⁽⁴⁾ using several hypotheses.* In Figure HEDL.4, we show the effective strain-time curves at an effective stress of 20,000 psi for virgin specimens as computed from the creep equation describing the pressurized tube results. We also present the current data for the virgin specimen at 850°F; the last measured strain at 850°F was 0.01%, although some additional strain recovery did occur on final unloading and heating to 1000°F. The present data at 1000°F are plotted in Figure HEDL.4 using two different assumptions: (a) the prior creep at 850°F has no effect, so the creep response is that of a virgin specimen, and (b) the effect of prior creep at 850°F is accounted for assuming a strain hardening rule. Both assumptions give reasonable agreement with predicted response of a virgin specimen, since the initial departure of the strain hardening assumption from the predicted curve can be rationalized as anelastic strain which would not have occurred if the specimen had not been unloaded at 850°F. However, for the assumption of virgin specimen behavior at 1100°F, the current results give strains a factor of 2 to 3 smaller than predicted values. The strain hardening assumption gives somewhat better agreement as shown in Figure HEDL.4, but the construction shown still predicts a strain value of 0.108% at 1000 hours for the present test, while we observed a strain of only 0.068%. We therefore conclude that, while prior strain history apparently has an effect on the 1100°F creep response, the simple strain hardening rule does not fully explain the observed decrease in creep rate over the first 1000 hours of testing.

Creep recovery results for all three test temperatures are presented in Figure HEDL.5. The time dependence of strain recovery is essentially the same at 850 and 1000°F, with the magnitude of strain recovery being about four times larger at 1000°F. On the other hand, the total strain recovery is comparable in magnitude at both 1000 and 1100°F, but the recovery occurs more rapidly at the higher temperature. The magnitude of creep strain recovery observed in these tests is only a small fraction of the elastic strain.

*Since creep curves for virgin specimens at 1000 and 1100°F were not obtained in the present work, it was necessary to use the pressurized tube results to represent creep behavior for virgin material. The pressurized tubes and the present specimen were made from the same heat of steel and are all 20% cold-worked, although the detailed fabrication history is somewhat different.

HEDL 7412-22.19

FIGURE HEDL.4. Creep Response of 20% Cold-Worked Type 316 Stainless Steel Under Varying Load and Temperature.

HEDL-7

FIGURE HEDL.5. Creep Recovery Results for 20% Cold-Worked Type 316 Stainless Steel at 850, 1000 and 1100°F.

HEDL 7412-22.20

The observed creep response after reloading is slightly different at the three test temperatures. Creep deformation after reloading is in good agreement with that predicted from the reoccurrence of anelastic strain and continued plastic creep strain at 850°F,⁽²⁾ but is slightly higher than predicted at 1000°F (Figure HEDL-2) and slightly lower than predicted at 1100°F (Figure HEDL-3). Our earlier view⁽²⁾ was that the reloading results at 1000°F suggested possible static thermal recovery during the prior unload period. However, in view of the fact that the 1100°F results show no evidence of enhanced creep strain after reloading, it now appears that static thermal recovery is not significant in these experiments. We presently have no physical explanation for the slight differences in observed and predicted response after reloading.

The microwave extensometer employed in this test has now operated for about 5600 hours over the temperature range from 850 to 1100°F. Strain measurement sensitivity is still on the order of 10^{-6} , and the reproducibility of the elastic extension and contraction measurements, which were made periodically throughout the test, indicate that the extensometer calibration has not changed perceptibly during service. Such results continue to demonstrate the applicability of microwave extensometers for sensitive, precise creep strain measurements.

3. Conclusions and/or Recommendations. Creep curves obtained from a single specimen of 20% cold-worked Type 316 stainless steel tested at 20,000 psi over a series of progressively higher temperatures (850, 1000, 1100°F) show that creep strain increases with increasing temperature. The introduction of only 0.06% creep strain during the 850 and 1000°F loadings appears to significantly affect the creep response at 1100°F. A simple strain hardening rule does not fully account for the effect of prior strain history. Creep response after unloading exhibits measurable strain recovery at all temperatures and is consistent with an anelastic component in the total creep deformation. The magnitude of recovered strain is of the order of 0.02% at both 1000 and 1100°F. To a first approximation, creep response after reloading is consistent with the reintroduction of anelastic strain recovered during the prior unload and the continued accumulation of creep strain as if unloading had not occurred. Test results continue to demonstrate the applicability of the microwave extensometer for sensitive, precise creep strain measurements.

B. EFFECT OF IRRADIATION ON THE MECHANICAL PROPERTIES OF 20% C.W. TYPE 316 STAINLESS STEEL PROTOTYPIC CLADDING

C. W. Hunter, G. D. Johnson and R. L. Fish, J. J. Holmes

1. Objective

The purpose of this work is to determine how neutron irradiation in EBR-II has altered the mechanical properties of FTR cladding material. These results provide information germane to the design, performance and safety analysis of fast reactor fuel pins and ducts.

2. Accomplishments and Status

a. Scope of Work. The neutron environment in a fast reactor alters the mechanical properties of the fuel pin cladding and duct material. Tensile tests on irradiated material were used to determine the short term strength and ductility at reactor refueling and operating temperatures. Stress-rupture tests indicated how the long-term loadbearing

capability was affected and simulated transient tests provided results germane to the performance and safety analysis of terminated and unterminated, possible and hypothetical fast reactor transients. The Larson-Miller parameter was employed to correlate the strength properties from the three types of tests.

b. Experimental Technique. All specimens were 2.45 inches long and had been sectioned from 0.230 inch O.D. x 0.015 inch wall tubing. The tubing material was 20% cold-worked Type 316 stainless steel (N-lot). These tubular specimens were placed in sodium-filled subcapsules to minimize temperature gradients during irradiation in the EBR-II. The subcapsules were positioned in a M-1A experimental pin to provide a predetermined helium gas gap. By utilizing gamma heating, the desired irradiation temperatures were obtained. Nominal temperatures of 860, 960 and 1050°F were obtained during irradiation to fluences of 3.6×10^{22} n/cm² (E > 0.1 MeV).

The tensile tests were performed on a hard beam testing machine with an Instron load-extension recorder. An LVDT extensometer was used to measure specimen elongation. Swagelok components were used to grip the specimens. Tests were performed at the irradiation temperature as well as at higher temperatures to simulate transient conditions. A vacuum furnace was used to obtain the desired test temperature with about 15 to 25 minutes for specimen heatup and thermal stabilization. An initial strain rate of 1.82×10^{-3} /min was used for all tensile tests.

The simulated transient tests were performed with the Fuel Cladding Transient Tester (FCTT) described in References 5-8. The specimens were rapidly heated under internal gas pressure loading until rupture occurred. Gas pressure of 6000 psi was used at 10°F/sec and at 200°F/sec, 4000 psi was employed. Both uniaxial and biaxial (internal gas pressure) stress-rupture tests were performed at 1000°F. The uniaxial tests were conducted under constant load conditions and the biaxial tests were performed at constant gas pressure as described in detail in Reference 9.

c. Tensile Test Results. The tensile test results consisting of Proportional Elastic Limit (PEL), Yield Strength (YS), Ultimate Tensile Strength (UTS), Uniform Elongation (UE), and Total Elongation (TE) are listed in Table HEDL.1 and illustrated in Figures HEDL.6 through .9. Figures HEDL.6 and .7 indicate the effect of test temperature on the strength and ductility. The data at 450°F are of interest to handling operations during refueling. The results at 1200°F are pertinent to a slow transient or other condition during which the temperature is elevated above the steady-state irradiation temperature.

At a test temperature of 450°F the Q specimens (3.6×10^{22} n/cm² at 860°F) are much stronger and have lower ductility than the FF specimens (1.3×10^{22} n/cm² at 960°F). Similar differences are also exhibited at the 900 and 1000°F test temperatures in Figures HEDL.6 and .7. This large difference results from greater neutron displacement damage from the higher fluence and lower irradiation temperature producing a harder crystal matrix in the Q specimens. At the 1200°F test temperature the properties for both irradiation conditions are very similar. Apparently, the heating up and hold times (15 to 25 minutes) for the 1200°F tests have produced sufficient annealing of the irradiation displacement damage, that the Q specimens no longer possessed a harder crystal matrix. This alteration of the irradiated microstructure by the heating and hold time above the irradiation

FIGURE HEDL.6. Effect of Test Temperature on Tensile Strength.

FIGURE HEDL.7. Effect of Test Temperature on Tensile Ductility.

TABLE HEDL.1
TENSILE TEST RESULTS

Specimen Identification	Irradiation Conditions		Test Conditions		Strength			Elongation	
	Fluence 10^{22} n/cm ² (E>0.1 MeV)	Temperature (°F)	Strain Rate 1/min	Temperature (°F)	PEL ksi	YS ksi	UTS ksi	UE %	TE %
Q13	3.6	860	1.82×10^{-3}	450	81.5	110.3	122.2	5.0	6.3
Q14	3.6	860	1.82×10^{-3}	900	58.4	94.8	104.6	2.4	2.9
Q16	3.6	860	1.82×10^{-3}	1200	25.6	43.6	50.4	1.5	2.8
FF4	1.3	960	1.82×10^{-3}	450	37.5	68.4	92.8	14.2	16.9
FF5	1.3	960	1.82×10^{-3}	1000	38.2	57.4	69.1	3.5	NA
FF6	1.3	960	1.82×10^{-3}	1200	31.5	49.2	53.4	1.75	3.3

NA - The total elongation value of specimen FF5 was inadvertently lost.

temperature illustrates the necessity for simulating the transient heating conditions (such as with the FCTT equipment) during the generation of mechanical properties for the performance and safety analysis of reactor transients.

Figures HEDL.8 and .9 indicate the effect of irradiation on the strength and ductility at test temperatures (900 and 1000°F) near the irradiation temperature. The low-fluence data in Figures HEDL.8 and .9 were taken from previous HEDL work. The results further substantiate that at the lower irradiation temperature (860°F) and higher fluence (3.6×10^{22} n/cm²), irradiation hardening has occurred.

d. Simulated Transient Tests. The results from the FCTT tests are listed in Table HEDL.2 and presented in Figures HEDL.10 through .12. For the same loading stress, the

TABLE HEDL.2
TRANSIENT BURST TESTS

Specimen Identification	Fluence 10^{22} n/cm ² (E>0.1 MeV)	Temperature (°F)	Gas Pressure psig	Hoop Stress ksi	Heating Rate °F/sec	Failure Temperature (°F)	Decrease in Failure Temp. (Δ°F)	Remaining Loadbearing Capability, %	Diametral Failure Strain, %
Q43	3.6	860	6000	43.2	10	1475	95	79.3	0.97
FF2	1.3	960	6000	43.2	10	1520	50	88.2	0.59
FF3	1.3	960	4000	28.8	200	1745	65	82.3	0.62
K1	3.4	1050	6000	43.2	10	1460	110	77.1	0.84
K2	3.4	1050	4000	28.8	200	1700	110	72.9	0.34

irradiated specimens failed at lower temperatures during the transient than did the unirradiated controls. Because of the strong temperature dependence of the failure stress curve in Figure HEDL-10, this decrease in the failure temperature indicates that the "strength" or loadbearing capability has been reduced.⁽⁷⁾ The decreased loadbearing capability apparently results from helium embrittlement.^(7,11,12) Figure HEDL.11 more clearly depicts the effect

FIGURE HEDL.8. Effect of Fluence on Tensile Strength at the Irradiation Temperature.

FIGURE HEDL.9. Effect of Fluence on Tensile Ductility at the Irradiation Temperature.

FIGURE HEDL.10. Failure Stress of Irradiated Specimens During Simulated Transient Tests.

FIGURE HEDL.11. Effect of Fluence on Loadbearing Capability During a Transient.

of neutron exposure on loadbearing capability. The decrease in ductility, resulting from helium embrittlement,^(7,11,12) is shown in Figure HEDL.12.

e. Stress-Rupture Results. The results for uniaxial stress-rupture tests and one biaxial test are listed in Table HEDL.3 and illustrated in Figures HEDL.13 and .14. As

TABLE HEDL.3
STRESS-RUPTURE RESULTS

Specimen Identification	Irradiation Conditions		Test Conditions		Results		
	Fluence 10^{22} n/cm ² (E>0.1 MeV)	Temperature (°F)	Stress ksi	Temperature (°F)	Time to Rupture (hr)	Total Creep Strain %	Minimum Strain Rate 1/hr
FF20	1.3	955	60	1000	226	4.6	1.38×10^{-4}
FF21	1.3	955	55	1000	269	3.6	2.82×10^{-5}
FF22	1.3	955	65	1000	106	6.2	3.81×10^{-4}
FF44	1.3	950	55*	1000	644	0.67	1.04×10^{-5}
K29	3.4	1050	60	1000	23	3.6	1.38×10^{-3}
K32	3.4	1050	52	1000	105	2.9	2.36×10^{-3}

*Biaxial test, internal gas pressure loading; the strain rate is the average strain rate.

indicated in Figure HEDL.13, the higher-fluence data exhibited a larger decrease in rupture life, and consequently a larger decrease in loadbearing capability. The failure strain is shown to decrease with fluence in Figure HEDL.14. Also, it is important to note the large differences in failure strain between the biaxial and uniaxial tests. The low fluence and unirradiated data in Figures HEDL.13 and .14 were taken from References 13 and 14.

f. Correlation of Results. Proper evaluation of the interaction of test time and temperature on the effects of neutron fluence on the mechanical properties of a material is sometimes difficult due to the limited amount of data. For example, the stress-rupture tests given in Table HEDL.3 were conducted at only one temperature. Proper assessment of the fluence effects at other temperatures would be impossible using these data alone. A correlation scheme for unifying various mechanical property data for 20% C.W. Type 316 stainless steel has been developed.⁽¹⁵⁾ Application of this correlation to the evaluation of the effects of neutron fluence on mechanical behavior was reported earlier.⁽¹⁶⁾

This correlation applies the life fraction rule and the Larson-Miller parameter to burst and transient data. Incorporation of a modulus-modified stress permits tensile data to be used in the correlation. Use of this correlation procedure yielded a relationship between the modulus-modified stress and the Larson-Miller parameter. Data for tensile, stress-rupture, burst and transient tests were reduced to a single curve described by $\sigma = f(\text{LMP})$.

Data presented in Tables HEDL.1 through .3 can be used to estimate the effects of neutron fluence for various imposed conditions. A summary of Larson-Miller parameters and modulus modified stresses is presented in Table HEDL.4 for the three types of tests conducted on the irradiated 20% C.W. Type 316 stainless steel tubing. These values are shown in Figure HEDL-15 with the unirradiated control curve from Reference 15. In Figure HEDL.15, the data

FIGURE HEDL.12. Comparison of Irradiated and Unirradiated Ductility.

FIGURE HEDL.13. Stress Rupture of 20% C.W. Type 316 Stainless Steel at 1000°F.

FIGURE HEDL.14. Rupture Strains for 20% C.W. Type 316 Stainless Steel at 1000°F.

FIGURE HEDL.15. Effect of Neutron Irradiation on the Failure Behavior of 20% C.W. Type 316 Stainless Steel Tubing.

TABLE HEDL.4
LARSON-MILLER PARAMETERS

Specimen Identification	Type of Test	Neutron Fluence 10^{22} n/cm ²	Applied or Ultimate Stress, ksi	Test or Failure Temp., °F	Modulus Modified Stress, ksi	Larson-Miller Parameter 10^{-4}
FF2	Transient	1.3	43.2	1520	45.5	3.40
FF3	Transient	1.3	28.8	1745	33.8	3.49
K1	Transient	3.4	43.2	1460	44.2	3.29
K2	Transient	3.4	28.8	1700	33.1	3.42
Q43	Transient	3.6	43.2	1475	44.7	3.32
FF5	Tensile	1.3	69.1	1000	64.8	2.87
FF6	Tensile	1.3	53.4	1200	49.1	3.23
Q14	Tensile	3.6	104.6	900	94.3	2.64
O16	Tensile	3.6	50.4	1200	46.4	3.22
FF20	Stress-Rupture	1.3	60	1000	50.7	3.28
FF21	Stress-Rupture	1.3	55	1000	46.4	3.35
FF22	Stress-Rupture	1.3	65	1000	54.8	3.22
FF44	Stress-Rupture	1.3	55	1000	46.4	3.33
K29	Stress-Rupture	3.4	60	1000	50.7	3.12
K32	Stress-Rupture	3.4	52	1000	43.9	3.22

from Table HEDL.4 lie on two curves, one for $\phi t = 1.3 \times 10^{22}$ n/cm², the other for $3.4\text{--}3.6 \times 10^{22}$ n/cm². Both of these curves are displaced below the unirradiated control curve, thus indicating a degrading effect of irradiation.

The curve for data from Reference 16 for $\phi t = 2.4\text{--}3.5 \times 10^{22}$ n/cm² is also shown in Figure HEDL.15. This curve is displaced from the unirradiated curve by a greater amount than the present data at similar fluences. The curve presented from Reference 16 was based on results from specimens taken from fuel pins. Thus, these fuel pin specimens had been subjected to fission gas loading, thermal gradients and different corrosion conditions while in EBR-II. The present specimens were unstressed and not in contact with mixed oxide fuel or flowing sodium.

3. Conclusions and Future Work

Results from tensile, stress-rupture and transient tests on irradiated 20% C.W. Type 316 stainless steel have been presented for neutron fluences up to 3.6×10^{22} n/cm² ($E > 0.1$ MeV). Particularly in the transient and stress-rupture tests, a degrading effect was found for increasing fluence. Failure data from these three types of tests were correlated with a Larson-Miller parameter.

Additional mechanical property tests will be performed on 60 irradiated tubing specimens. Results from these tests will be incorporated into the Larson-Miller correlation presented in Figure HEDL-15. This will provide a data base for determining the effect at neutron fluence on the mechanical properties of 20% C.W. Type 316 stainless steel.

C. TEST TEMPERATURE AND NOTCH EFFECTS ON THE TENSILE BEHAVIOR OF HIGHLY IRRADIATED
EBR-II DUCT THIMBLE MATERIAL

R. L. Fish, C. W. Hunter, J. D. Watrous, J. J. Holmes

1. Objective

The objective of this work is to develop an understanding of how notches, strain rate and temperature affect the tensile deformation and failure of highly irradiated stainless steel. This information obtained from high-fluence annealed Type 304 stainless steel will aid in the extrapolation of present low-fluence 20% C.W. Type 316 properties.

2. Accomplishments and Status

a. Irradiation Conditions. EBR-II irradiated Type 304 stainless steel duct thimbles used in this work and their associated irradiation conditions are summarized in Table HEDL.5.

TABLE HEDL.5
SUMMARY OF EBR-II IRRADIATED DUCT THIMBLES

EBR-II Thimble Identification	Exposure (Mwd)	Peak Fluence ($E > 0.1$ MeV)	Irradiation Temp. ($^{\circ}$ F)
3A1 SRT	37,490	11.7×10^{22} n/cm ²	700-860
5A3 CRT	36,785	7.6×10^{22} n/cm ²	700-870
5C3 CRT	15,541	3.7×10^{22} n/cm ²	700-870
5F3 CRT	42,321	10.3×10^{22} n/cm ²	700-870
5F1 CRT	49,967	10.2×10^{22} n/cm ²	700-870

Irradiation temperatures for specimens sectioned from the thimbles were estimated from curves supplied by R. D. Phipps of the EBR-II project. The fluence estimates were generated by J. A. Ulseth and J. L. Jackson of the dosimetry center at HEDL.

A plot of the estimated irradiation conditions for one of the thimbles, control rod thimble (CRT) 5A3, is shown in Figure HEDL.16. The axial neutron flux gradient in the EBR-II is quite steep because of the relatively short fuel column length (13.5 inches). The neutron fluences for the components are thus about three times higher at the midplane than at 10 inches away from the midplane. The temperature gradients vary from flat to flat, depending upon the loading of adjacent subassemblies. However, in general, at distances greater than 6-3/4 inches below the core center, the thimble temperature is equal to the inlet sodium coolant temperature, which is maintained at a constant 700°F. The temperature rises fairly rapidly through the core region and becomes approximately constant again for distances more than about 10 inches above the midplane. The temperature profiles in Figure HEDL.16 represent average profiles over the lifetime of in-reactor service.

b. Tensile Testing. Tensile tests were performed on a hard beam Instron testing machine equipped with a high temperature vacuum furnace. Both load and elongation were recorded throughout the test. An LVDT extensometer capable of 700X strain magnification was employed for strain measurements during the low strain (<1.0%) portion of all tests. For the specimens exhibiting very low plastic strain, the extensometer recorded the entire elongation history for the test. The elongation on specimens exhibiting more extensive strain

FIGURE HEDL.16. Estimated Irradiation Conditions for Control Rod Thimble 5A3.

was estimated by summing the extensometer and, beyond yielding, chart drive and crosshead movement correlations. Tests were performed over the initial strain rate range of 2.67×10^{-3} /minute to 2.67/minute. The specimen temperature during each test was measured and controlled with a chromel-alumel thermocouple. Approximately 20 to 30 minutes were allowed for temperature stabilization prior to initiating each tensile test. All tests were performed on material irradiated near 700°F.

The "smooth" specimen design used in this work is shown in Figure HEDL.17. It is a modification of the pin-loaded flat specimen described in ASTM Standard E8. The overall dimensions were 2-5/8 inches long by 0.040 inch thick; the reduced gage section was 3/4 inch long by 1/8 inch wide. The notched specimens were fabricated by machining the notches on one side of the smooth specimen to a depth of 0.030 inch. Two notch geometries were investigated, 0.003 inch radius and 0.030 inch radius.

c. Smooth Specimen Results. The tensile test results generated using smooth specimens are presented in Table HEDL.6 along with the respective specimen irradiation temperatures and fluences. Also included in the table are test temperature and strain rate. The major groupings within the table are based on test temperature.

d. Effect of Test Temperature. The tensile properties listed in Table HEDL.6 for the very high fluence ($>8.7 \times 10^{22}$ n/cm²) material at the nominal strain rate of 2.67×10^{-3} /minute are plotted as a function of test temperature in Figures HEDL.18 and .19. The yield strength (0.2% offset) and ultimate tensile strength are shown in Figure HEDL.18. At these very high fluences (8.7 - 10.3×10^{22} n/cm²) the strength parameters continually decrease with increasing test temperature from room temperature to 1400°F. At temperatures above 1000°F the strength drops more rapidly; concurrently, the plastic strain to maximum load decreases to below the 0.2% level rendering the 0.2% offset yield strength parameter meaningless above this temperature. The ultimate tensile strength becomes less temperature dependent at 1300°F and above.

The uniform and total elongation at the high fluences are plotted as a function of test temperature in Figure HEDL.19. These ductility parameters decrease with increasing temperature between room temperature and 450°F. Between 450 and 700°F the parameters are temperature independent with uniform elongation at ~0.6% and total elongation at about the 1.8% level. As temperature is increased from 900 to 1100°F the ductility decreases rapidly, becoming very low and temperature independent above 1100°F. At these high temperatures ($\geq 1100^\circ\text{F}$) uniform and total elongation are identical, because essentially no plastic strain occurs beyond the peak load.

e. Strain Rate Effects. At the lower test temperatures of room temperature, 450 and 700°F, strain rate effects were investigated. The ultimate tensile strengths measured at these temperatures are plotted as a function of strain rate in Figure HEDL-20. The strain rate dependence, although not strong at any temperature investigated, tended to be independent of test temperature. A slight increase in ultimate tensile strength of about 8% was observed as the strain rate increased from 2.67×10^{-3} /minute to 2.67/minute.

FIGURE HEDL.17. EBR-II Control and Safety Rod Thimble Tensile Specimen.

FIGURE HEDL.18. Effect of Test Temperature on the YS (0.2% Offset) and UTS at High Fluence.

TABLE HEDL.6

TENSILE PROPERTIES OF FAST NEUTRON IRRADIATED, ANNEALED TYPE 304 STAINLESS STEEL

Specimen Identification	Strain Rate (1/min)	Irrad. Temp. (°F)	Fluence E>0.1 MeV (n/cm ²)	Test Temp. (°F)	Strength, ksi			Ductility, %		
					PEL	YS	UTS	UE	TE	RA
C1A1	2.67x10 ⁻³	700	3.8x10 ²²	R.T.	101.4	126.8	132.0	4.1	11.8	50.0
C2A1	2.67x10 ⁻²	700	3.5x10 ²²	R.T.	92.3	119.1	125.0	7.0	12.1	53.2
C5A1	2.67x10 ⁻¹	700	3.5x10 ²²	R.T.	96.1	118.4	123.7	4.5	9.0	56.6
C6A1	2.67	700	3.8x10 ²²	R.T.	110.2	138.8	140.6	4.9	11.1	54.9
C1C1	2.67x10 ⁻³	700	7.8x10 ²²	R.T.	102.3	133.3	141.4	2.2	6.0	47.9
C2C1	2.67x10 ⁻²	700	7.2x10 ²²	R.T.	103.6	135.2	143.6	4.5	11.2	50.0
C5C1	2.67x10 ⁻¹	700	7.2x10 ²²	R.T.	104.3	137.6	148.0	6.1	12.0	45.2
C6C1	2.67	700	7.8x10 ²²	R.T.	109.6	139.1	145.1	2.4	6.8	45.4
C1E1	2.67x10 ⁻³	730	10.3x10 ²²	R.T.	84.6	127.1	137.9	2.8	4.6	26.6
C2E1	2.67x10 ⁻²	730	9.4x10 ²²	R.T.	94.7	129.8	140.2	4.0	6.6	28.4
C3E1	2.67x10 ⁻¹	730	8.8x10 ²²	R.T.	108.2	136.9	147.6	5.0	10.8	47.5
C6E1	2.67	730	10.3x10 ²²	R.T.	101.9	135.8	149.1	5.2	7.2	17.0
C1A2	2.67x10 ⁻³	700	3.8x10 ²²	450	75.0	105.2	106.7	0.7	3.3	37.2
C2A2	2.67x10 ⁻²	700	3.5x10 ²²	450	84.3	109.3	111.0	1.0	3.8	31.4
C5A2	2.67x10 ⁻¹	700	3.5x10 ²²	450	95.7	117.0	119.6	0.9	4.8	40.1
C6A2	2.67	700	3.8x10 ²²	450	95.6	125.4	126.9	1.1	3.7	44.3
C1C2	2.67x10 ⁻³	700	7.8x10 ²²	450	90.9	116.6	118.0	0.5	2.5	41.4
C2C2	2.67x10 ⁻²	700	7.2x10 ²²	450	99.8	127.1	131.2	1.1	3.6	34.9
C5C2	2.67x10 ⁻¹	700	7.2x10 ²²	450	93.4	124.3	129.1	1.0	2.8	48.9
C6C2	2.67	700	7.8x10 ²²	450	102.2	134.8	139.3	1.0	2.0	37.0
C1E2	2.67x10 ⁻³	730	10.3x10 ²²	450	90.0	120.8	123.6	0.6	1.9	24.0
C2E2	2.67x10 ⁻²	730	9.4x10 ²²	450	100.0	129.3	133.8	1.2	2.9	26.8
C3E2	2.67x10 ⁻¹	730	8.8x10 ²²	450	97.1	128.9	134.1	1.2	2.9	38.0
C6E2	2.67	730	10.3x10 ²²	450	100.0	130.4	133.3	1.0	1.7	20.4
B6D1	2.67x10 ⁻³	700	2.8x10 ²²	700	74.1	106.7	108.5	0.52	2.2	30.1
B6E1	2.67x10 ⁻²	700	4.5x10 ²²	700	73.0	110.1	112.6	0.48	2.0	22.7
B6F1	2.67x10 ⁻³	700	6.6x10 ²²	700	81.9	112.6	115.7	0.5	3.3	21.5
D2E2	2.67x10 ⁻³	725	9.7x10 ²²	700	93.1	105.9	106.0	0.6	1.7	24.5
D3E2	2.67x10 ⁻²	725	9.2x10 ²²	700	84.0	110.6	114.4	0.8	1.6	25.2
D4E2	2.67x10 ⁻¹	725	9.7x10 ²²	700	88.5	109.0	115.8	1.0	1.5	31.5
D5E2	2.67	725	10.2x10 ²²	700	84.6	111.2	115.8	0.9	1.4	26.6
H06451 (5C3)	0.05	704	0.93x10 ²²	1100	44.3	60.2	60.6	0.9	2.0	30.6
H06461 (5C3)	0.05	704	1.6x10 ²²	1100	44.9	60.4	60.4	1.0	1.1	11.4
H06493 (5C3)	0.05	750	3.0x10 ²²	1100	43.4	56.3	56.3	0.4	0.5	7.9
B6B1	2.67x10 ⁻³	700	1.1x10 ²²	1100	43.3	55.2	57.5	0.77	1.8	17.3
B6C1	2.67x10 ⁻²	700	1.8x10 ²²	1100	57.1	67.3	68.0	0.36	1.1	19.2
A5A2	2.67x10 ⁻³	700	2.8x10 ²²	1100	65.7	----	73.5	0.18	0.6	24.1
A5C2*	2.67x10 ⁻³	700	5.85x10 ²²	1100	50.9	----	53.0	0.01	0.01	----
A5E2*	2.67x10 ⁻³	730	7.56x10 ²²	1100	41.9	----	42.1	0.01	0.2	----
H06455 (5C3)	0.05	704	0.93x10 ²²	1300	14.6	21.1	26.6	5.6	9.6	18.3
H06463 (5C3)	0.05	704	1.6x10 ²²	1300	21.5	34.9	35.8	0.8	0.9	11.1
H06494 (5C3)	0.05	750	3.0x10 ²²	1300	19.1	30.8	30.8	0.25	0.3	9.4
D1D1	2.67x10 ⁻³	710	9.7x10 ²²	1000	69.8	81.3	81.3	0.2	0.40	10.0
D2D1*	2.67x10 ⁻²	710	9.2x10 ²²	1100	51.2	----	56.4	0.04	0.04	----
D3D1	2.67x10 ⁻³	710	8.7x10 ²²	1200	31.4	----	38.1	0.04	0.06	3.3
D4D1*	2.67x10 ⁻³	710	9.2x10 ²²	1300	22.9	----	27.7	0.04	0.07	----
D5D1	2.67x10 ⁻³	710	9.7x10 ²²	1400	19.4	----	23.7	0.04	0.06	4.6

*Broke at extensometer point.

A-5A3 CRT
 B-3A1 SRT
 C-5F3 CRT
 D-5F1 CRT

FIGURE HEDL.19. Effect of Test Temperature on the Elongation at High Fluence.

FIGURE HEDL.20. Effect of Strain Rate on the Ultimate Tensile Strength at High Fluence.

The uniform elongation at the high fluence levels ($8.8-10.3 \times 10^{22} \text{ n/cm}^2$) is plotted as a function of strain rate in Figure HEDL.21. The strain rate dependence of uniform elongation is more pronounced than that observed in the strength. Particularly at room temperature the uniform elongation increases substantially as the strain rate increases from $2.67 \times 10^{-3}/\text{minute}$ to $2.67/\text{minute}$.

f. Effect of Fluence. The effect of fluence on the ultimate tensile strength of annealed Type 304 stainless steel is presented in Figure HEDL.22. At test temperatures less than and equal to the 700°F irradiation temperature, the strength increases rapidly with increasing fluence until about $3 \times 10^{22} \text{ n/cm}^2$ above which the fluence dependence weakens. Above $\sim 7 \times 10^{22} \text{ n/cm}^2$ the strength levels out and becomes fluence independent. The low fluence strength representations shown in Figure HEDL.22 were established on the basis of previously published data at 700°F ⁽¹⁷⁾ and assuming similar trends for the 450°F and room temperature curves. At the 1100 and 1300°F test temperatures, ultimate tensile strength exhibited less of a fluence dependence than at the lower temperatures. The strength increases with fluence to about $3 \times 10^{22} \text{ n/cm}^2$ above which the strength drops abruptly and becomes independent of fluence. At all fluence levels investigated strength decreased with increasing test temperature.

Total elongation measured at and below the 700°F irradiation temperature is shown as a function of fluence in Figure HEDL.23. Over the fluence range investigated, 3 to $10 \times 10^{22} \text{ n/cm}^2$, the total elongation at 700°F remained fluence independent. There is a greater fluence dependence at test temperatures below 700°F . Both the uniform and total elongation at 1100 and 1300°F are presented as a function of fluence in Figure HEDL.24. These ductility parameters decrease rapidly with increasing fluence to about $4 \times 10^{22} \text{ n/cm}^2$. The total plastic strain to failure above $4 \times 10^{22} \text{ n/cm}^2$ corresponds very closely with the plastic strain at the peak load. Therefore, uniform and total elongation become essentially identical.

g. Notched Specimen Results. The tensile test results on notched specimens are presented in Table HEDL.7. The irradiation conditions, test temperature, strain rate, and specific notch geometry are listed in the table also. The data are grouped according to test temperature.

h. Effect of Test Temperature. The sharpest notch investigated (0.003 inch radius) resulted in a definite notch effect, which is illustrated in Figure HEDL.25. In this figure the ultimate tensile strength has been plotted as a function of test temperature. All data on this plot were taken from specimens irradiated to an intermediate fluence range (3.8 to $5.85 \times 10^{22} \text{ n/cm}^2$). Unnotched material ultimate tensile strength is shown as a solid line. The dashed line represents the notched material behavior. All strength values are based upon the net cross-sectional stress. A significant notch strengthening exists at room temperature and a notch sensitivity or weakening at 1100°F . No significant notch effect was observed at 450 and 700°F . Figure HEDL.26 illustrates the effect of notches on the ultimate tensile strength at the very high fluences (8.7 to $10.3 \times 10^{22} \text{ n/cm}^2$). Strength is plotted as a function of test temperature with the solid line again representing the unnotched material behavior. The notch sensitivity at both the intermediate and high fluence levels effected a nominal 30% decrease in ultimate tensile strength from the unnotched level.

FIGURE HEDL.21. Effect of Strain Rate on the Uniform Elongation at High Fluence.

FIGURE HEDL.22. Effect of Fluence on the Ultimate Tensile Strength.

FIGURE HEDL.23. Effect of Fluence on Total Elongation.

FIGURE HEDL.24. Effect of Fluence on the High Temperature Elongation.

FIGURE HEDL.25. Notch Effect on the Ultimate Tensile Strength at Intermediate Fluence.

FIGURE HEDL.26. Notch Effect on the Ultimate Tensile Strength at High Fluence.

TABLE HEDL.7

TENSILE TEST DATA ON NOTCHED EBR-II THIMBLE SPECIMENS

Specimen Ident.	Notch Geometry	Strain Rate (1/min)	Irrad. Temp. (°F)	Fluence E>0.1 MeV (n/cm ²)	Test Temp. (°F)	Strength, ksi			Ductility, %		
						PEL	YS	UTS	UE	TE	RA
C1B1	.030Dx.003R	2.67x10 ⁻³	700	5.6x10 ²²	1100	37.1	-----	41.0	0.04	0.07	10.6
C2B1	.030Dx.003R	2.67	700	5.1x10 ²²	1100	60.9	69.7	71.6	0.4	0.4	16.9
C5B1	.030Dx.030R	2.67x10 ⁻³	700	5.1x10 ²²	1100	30.6	-----	35.0	0.05	0.07	2.8
C6B1	.030Dx.030R	2.67	700	5.6x10 ²²	1100	60.0	85.6	89.2	0.4	0.5	4.0
C1D1	.030Dx.003R	2.67x10 ⁻³	710	9.7x10 ²²	1100	32.1	-----	35.7	0.04	0.07	5.0
C2D2	.030Dx.003R	2.67	710	8.9x10 ²²	1100	55.4	68.7	69.0	0.2	0.3	9.8
C5D1	.030Dx.030R	2.67x10 ⁻³	710	8.9x10 ²²	1100	31.8	-----	37.6	0.05	0.1	11.8
C6D1	.030Dx.030R	2.67	710	9.7x10 ²²	1100	71.9	92.2	93.1	0.3	0.3	7.4
C1B2	.030Dx.003R	2.67x10 ⁻³	700	5.6x10 ²²	R.T.	130.0	158.3	161.5	0.5	1.5	60.0
C2B2	.030Dx.003R	2.67x10 ⁻³	700	5.1x10 ²²	R.T.	78.0	131.5	154.5	1.7	1.9	36.3
C1D2	.030Dx.003R	2.67x10 ⁻³	710	9.7x10 ²²	R.T.	85.0	130.0	136.0	0.6	2.2	29.6
D1B1	.030Dx.003R	2.67x10 ⁻³	700	4.7x10 ²²	450	83.3	103.3	114.6	0.8	1.5	27.6
D2D2	.030Dx.003R	2.67x10 ⁻³	710	9.2x10 ²²	450	108.2	-----	122.0	0.15	0.16	13.6
D1B2	.030Dx.003R	2.67x10 ⁻³	700	4.7x10 ²²	700	85.4	108.3	108.5	0.25	0.8	18.8
D1E2	.030Dx.003R	2.67x10 ⁻³	725	10.2x10 ²²	700	83.6	-----	106.6	0.07	0.07	9.2

A-5A3 CRT
B-3A1 SRT
C-5F3 CRT
D-5F1 CRT

1. Effect of Geometry and Strain Rate on Notch Sensitivity. To investigate the notch sensitivity further, two different notch geometries were incorporated and two different strain rates used in performing tests at 1100°F. Figures HEDL.27 and .28 are plots of the ultimate tensile strength and elongation values as a function of fluence for these tests.

The ultimate tensile strength is shown in Figure HEDL.27 as a function of fluence for both 2.67 and 2.67 x 10⁻³/minute strain rate conditions and both 0.003 inch and 0.030 inch radii notch geometries. All tests were performed at 1100°F. The unnotched specimen ultimate tensile strength for these irradiation and test conditions (2.67 x 10⁻³/minute strain rate only) is represented by the dashed line. No fluence dependence was observed in this property for either of the strain rates or notch geometries over the fluence range investigated. At the lower strain rate (2.67 x 10⁻³/minute) the notch sensitivity did not reflect a dependence on notch acuity for the two geometries investigated. The ultimate tensile strength was measured at about 37 ksi for both geometries at this strain rate. However, the higher strain rate (2.67/minute) resulted in a notch geometry effect. The specimens with the larger 0.030 inch radius notch exhibited an ultimate tensile strength of about 90 ksi while the smaller 0.003 inch radius notch resulted in an ultimate tensile strength of only about 70 ksi.

Uniform and total elongation are shown in Figure HEDL.28 as a function of fluence for the two strain rates and notch geometries. These tensile parameters are not comparable to those measured using an unnotched specimen because most of the strain occurs in the notched region as opposed to a more uniform distribution of deformation in a smooth specimen. However, trends in ductility behavior may be illustrated with these parameters. The low strain rate (2.67 x 10⁻³/minute) elongation parameters exhibited no fluence dependence over

HEDL 7412-13.6

FIGURE HEDL.27. Effect of Strain Rate and Notch Geometry on the Ultimate Tensile Strength at High Fluence.

HEDL 7412-13.1

FIGURE HEDL.28. Effect of Strain Rate and Notch Geometry on the Uniform and Total Elongation at High Fluence.

the range investigated. A notch geometry effect was not evident at the lower strain rate. At the higher strain rate (2.67/minute) the uniform and total elongation did show a fluence dependence, decreasing as the fluence increased. Elongation was not significantly affected by the notch acuity variation at this strain rate either. The higher strain rate resulted in higher measures of ductility as illustrated in Figure HEDL.28.

j. Fractography

Fracture surfaces from several tensile specimens were examined using a scanning electron microscope (SEM). A composite SEM fractograph was developed for each specimen fracture using 75X magnification photos. This composite was used for locating higher magnification photos which were taken at up to 3000X. The composite also helped establish keys to low magnification (75-300X) appearance of fracture mechanisms identified and confirmed at the higher magnifications (1000-3000X). Using a knowledge of low magnification appearance of fracture mechanisms and confirming observations with the high magnification fractographs, an engineering judgment was made of the fracture surface percentage for each of three different types of fracture. This was done for each specimen. The results of these judgments are summarized in Table HEDL.8. Some tensile specimens from other Type 304 stainless steel studies were analyzed and have been included in this table in order to facilitate overall interpretation.

TABLE HEDL.8
SUMMARY OF SCANNING ELECTRON MICROSCOPE (SEM) FRACTOGRAPH ANALYSIS

Specimen Identification	Irrad. Temp., °F	Est. Fluence (E>0.1 MeV), n/cm ²	Test Temp., °F	Strain Rate, 1/min	Est. Percent of Fracture Surface		
					Dimpling	Channel	Intergran.
C1A1	700	3.8×10^{22}	R.T.	2.67×10^{-3}	80%	20%	0%
C1E1	730	10.3×10^{22}	R.T.	2.67×10^{-3}	3	97	0
C6E1	730	10.3×10^{22}	R.T.	2.67	0	100	0
C1A2	700	3.8×10^{22}	450	2.67×10^{-3}	85	15	0
C1E2	730	10.3×10^{22}	450	2.67×10^{-3}	0	100	0
C6E2	730	10.3×10^{22}	450	2.67	0	100	0
6A1	700	0.9×10^{22}	700	5.0×10^{-2}	100	0	0
A5A1	700	2.8×10^{22}	700	2.67×10^{-3}	70	30	0
A5C1	700	5.9×10^{22}	700	2.67×10^{-3}	30	70	0
A5E1	730	7.6×10^{22}	700	2.67×10^{-3}	4	96	0
B6H1	750	10.7×10^{22}	700	2.67×10^{-3}	0	100	0
D5E2	725	10.2×10^{22}	700	2.67	0	100	0
A5H1	840	2.8×10^{22}	800	2.67×10^{-3}	20	78	2
B6I1	795	9.4×10^{22}	800	2.67×10^{-3}	1	98	1
A5J1	870	0.76×10^{22}	900	2.67×10^{-3}	95	0	5
A5I1	860	1.8×10^{22}	900	2.67×10^{-3}	50	30	20
B6J2	840	6.6×10^{22}	900	2.67×10^{-3}	0	80	20
ALW4	---	0.0×10^{22}	1000	2×10^{-3}	99	0	1
A5E2	730	7.5×10^{22}	1100	2.67×10^{-3}	5	5	90
D2D1	710	9.2×10^{22}	1100	2.67×10^{-3}	10	65	25
ALW5	---	0.0×10^{22}	1200	2×10^{-3}	20	0	80
D5D1	710	9.7×10^{22}	1400	2.67×10^{-3}	1	0	99
C1D1*	710	9.7×10^{22}	1100	2.67×10^{-3}	5	0	95
C2D2*	710	8.9×10^{22}	1100	2.67	20	30	50
C5D1**	710	8.9×10^{22}	1100	2.67×10^{-3}	5	0	95

*Tensile notch 0.003 in. radius (fracture percent judgement near notch root).

**Tensile notch 0.030 in. radius (fracture percent judgement near notch root).

The fracture mechanisms observed in this material were transgranular plastic dimpling, channel fracture and intergranular fracture. The transgranular plastic dimpling and intergranular fracture are classical mechanisms of fracture observed in this material. The channel fracture mechanism which has been recently observed in irradiated stainless steel⁽²¹⁾ can be described as resulting from the confinement of slip to narrow bands of planes or channels within the crystal structure. At high fluences the slip along these bands becomes so extensive that the crystals literally slide apart. The resulting fracture surface appears relatively flat with many ledges interrupting the parallel faceted areas.

k. Correlation of Properties with Fracture Mechanism

The correlation of fracture mechanisms with tensile properties promotes a better understanding of the material behavior. The relationship between fracture and ultimate tensile strength as a function of fluence at two different test temperatures is shown in Figure HEDL.29. The low temperature (700°F) fracture is transgranular in nature at all fluence levels. However, the classical irradiation hardening, which is very fluence dependent at low fluence levels and becomes relatively fluence independent above 3×10^{22} n/cm², is accompanied by a transition in the fracture mechanism. At low fluences fracture occurs by plastic dimpling. As the fluence increases the shear processes cease to be homogeneous throughout the entire crystal and become restricted to the extent that complete channel fracture results at the highest fluence levels. At the 1100°F test temperature, fracture occurs at the low fluences by a mixed plastic dimpling and intergranular fracture. As fluence increases the tensile strength increases. Above about 3×10^{22} n/cm² the grain boundary embrittlement due to helium becomes effective enough to interfere with the loadbearing capability of the material and the tensile strength drops abruptly. The high fluence 1100°F fracture (see Figure HEDL.29) reflects a greater degree of intergranular failure, indicative of helium embrittled grain boundaries.

No fundamental difference in fracture mechanism was observed at R.T. to 700°F ($\sim 10^{23}$ n/cm², $E > 0.1$ MeV) due to the higher strain rate (2.67/minute) as illustrated in Figure HEDL.30. Channel fracture was observed to be the dominant mechanism at all temperatures (R.T., 450 and 700°F) and at both strain rates (2.67 and 2.67×10^{-3} /minute). It is presumed therefore that this mechanism is operative over these entire strain rate and temperature ranges at the high fluence level ($\sim 10^{23}$ n/cm², $E > 0.1$ MeV). The relationship between fracture and elongation at the very high fluence level ($\sim 10^{23}$ n/cm², $E > 0.1$ MeV) is presented in Figure HEDL.31 as a function of test temperature. In general, the continual decrease in both uniform and total elongation with increasing test temperature is associated with a decreasing tendency for flow within the grains. It is hypothesized that there are more channels operating (greater channel density) under the room temperature test conditions which results in the greater measured ductilities. Transmission electron microscopy (TEM) is planned to help investigate this possibility. The channel fracture observed at the lower temperatures (R.T. to 700°F) results in relatively high elongation values compared with the higher test temperature behavior.

The further decrease in elongation at the high temperatures is associated with the interference of matrix deformation by intergranular fracture as shown in Figure HEDL.31. The combination at high fluences of a strong matrix and helium embrittled grain boundaries allows

FIGURE HEDL.29. Fracture Mode and Irradiated Tensile Strength.

S.R.=2.67/MIN

ROOM TEMPERATURE

450 °F

700 °F

S.R.=2.67 x 10⁻³/MIN

ROOM TEMPERATURE

450 °F

700 °F

**IRRADIATION TEMP.=725-750 °F;
FLUENCE = 10.2-10.7 x 10²² n/cm² (E>0.1 MeV)**

FIGURE HEDL.30. Channel Fracture in High Fluence Type 304 Stainless Steel.

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FIGURE HEDL.31. Effect of Test Temperature on Ductility and Fracture.

initiation and propagation of an intergranular crack at very low plastic strain levels. Propagation of an intergranular crack precludes the possibility of further extensive deformation within the matrix of the material.

1. Fracture Map. The nineteen fracture surfaces listed in Table HEDL.8 from tensile specimens conducted at $\sim 10^{-3}$ /minute strain rate and evaluated with the aid of a SEM provide a basis for mapping the fracture surface character of annealed Type 304 stainless steel as a function of fast fluence and test temperature. Although it would be desirable to use many more fracture surface evaluations in generating the fracture character map, a reasonable description is felt possible at this time.

The onset and completion of the channel fracture mechanism were established on the basis of plotting fracture surface percent of this mechanism as a function of fast fluence as shown in Figure HEDL.32. Each fracture surface ($\leq 1100^{\circ}\text{F}$) displaying channel fracture was used in generating this plot. Note that a certain degree of scatter exists for the $700\text{--}900^{\circ}\text{F}$ data points. The scatter band for this temperature range was used to help establish bands for room temperature to 450°F and 1100°F temperatures. The intersection of the scatter bands with the zero channel fracture level established the "onset of channel fracture" zone presented on the fracture map in Figure HEDL.33. The intersection of the $700\text{--}900^{\circ}\text{F}$ scatter band with the 100% channel fracture level established the "completion of channel fracture transition" zone for that temperature range as shown in Figure HEDL.33. Data points (C1E1 and C1E2) for R.T. and 450°F temperatures indicate that the completion of channel fracture occurs near the same fluence level for these temperatures also. The "completion of channel fracture transition" zone is dashed above about 800°F to indicate a suspected trend only.

The onset of intergranular fracture was established using primarily four tensile specimens (A5H1, B6I1, A5J1 and ALW 4) which show minimal intergranular fracture characters. The unirradiated specimen (ALW 4) was tested at 1000°F . A specimen at $\sim 0.7 \times 10^{22} \text{ n/cm}^2$ (A5J1) was tested at 900°F and the other two at higher fluences tested at 800°F . The very low intergranular character of these fractures ($\leq 5\%$ in all cases), which display a decrease in test temperature with increasing fluence, tends to support about a 200°F shift in onset of intergranular fracture due to fast neutron irradiation of this material. This shift is represented by a zone of arbitrary width in Figure HEDL.33. Additional evidence for the existence of the shift in onset of intergranular fracture due to irradiation is the set of tensile fracture surfaces evaluated at 900°F (B6J2, A5I1, and A5J1). As can be observed in Table HEDL.8, the higher fluence 900°F specimens exhibited a factor of four greater degree of intergranular fracture character, as measured by percent of fracture surface, than the lowest fluence specimen. A lack of an adequate number of high temperature ($\geq 1200^{\circ}\text{F}$) tensile fracture surfaces rendered determination of the "completion of intergranular fracture transition" zone impossible. A dashed line is shown in Figure HEDL.33 to merely represent an anticipated trend for the near completion of the intergranular fracture transition.

m. Notched Specimen Fractography. Fracture surface characterization was performed on three notched tensile specimens also (C1D1, C2D2, and C5D1). The fracture character data are listed at the bottom of Table HEDL.8 for these specimens. No significant difference in fracture character (95% intergranular) was observed in the two low strain rate (2.67×10^{-3} /minute) notched specimens despite the factor of ten difference in notch radius (0.003 versus 0.030 inch). This observation is consistent with the similar strength and ductility

FIGURE HEDL.32. Effect of Fluence of Percent of Channel Fracture.

HEDL 7412-13.2

HEDL 7412-13, 14

FIGURE HEDL.33. Fluence-Test Temperature Fracture Map for EBR-II 304 Stainless Steel.

behavior exhibited at the low strain rate as shown in Figures HEDL.27 and .28. However, the notched (0.003 inch radius) specimen tested at the higher strain rate of 2.67/minute (C2D2) revealed substantially greater transgranular character (50%) than the low strain rate specimen with the same notch acuity. The higher elongation values exhibited at the high strain rate (see Figure HEDL.28) are consistent with the greater extent of transgranular fracture revealed by Specimen C2D2.

n. Conclusions. The tensile test results described in this report lead to the following conclusions relative to annealed Type 304 stainless steel:

- (a) The strength increases and elongation decreases rapidly with fluence to about 3×10^{22} n/cm² ($E > 0.1$ MeV).
- (b) In general, the strength and elongation properties become relatively fluence independent beyond $\sim 4 \times 10^{22}$ n/cm² ($E > 0.1$ MeV).
- (c) The strength and elongation properties both generally decrease with increasing test temperature at all fluence levels.
- (d) The tensile properties are moderately strain rate sensitive in the range of 2.67×10^{-3} to 2.67/minute at R.T. to 700°F ($\dot{\epsilon} \sim 10^{23}$ n/cm²).
- (e) This material is notch sensitive at 1100°F at fluences beyond $\sim 4 \times 10^{22}$ n/cm².
- (f) Notch acuity (0.003 inch to 0.030 inch radius) does not affect the notch sensitivity at a nominal tensile strain rate of 2.67×10^{-3} /minute, but does at the higher strain rate.

No further testing is presently planned on this material. Limited examination of previously tested specimens using a transmission electron microscope will be conducted to help clarify deformation in the transgranular fracture region.

D. THERMAL CREEP IN PRESSURIZED TUBES OF 20% COLD-WORKED TYPE 316 STAINLESS STEEL

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1. Objective

The purpose of this work is to determine the thermal creep deformation of pressurized tubes of 20% cold-worked Type 316 stainless steel. Results of these experiments will be compared with companion in-reactor tests to define the irradiation creep component of in-reactor deformation.

2. Accomplishments and Status

a. Scope of Work. Experimental measurements of creep strains in pressurized tube samples of 20% cold-worked Type 316 stainless steel were reported previously.⁽²²⁾ These specimens covered the temperature range from 825 to 1400°F for times to 2400 hours at stresses up to 60,000 psi. In this report, we present the results of a detailed analysis of these data which yield an analytical description of the experimental results. This description of thermal creep behavior finds application in the analysis and interpretation of in-reactor creep experiments, and also in inelastic analysis of reactor components.

Experimental details were reported previously,⁽²²⁾ but several features will be repeated here for clarity. We computed stresses using the thin-wall approximations:

$$\sigma_{\theta} = \frac{p d_m}{2w}$$

$$\sigma_z = \frac{p d_m}{4w}$$

$$\sigma_r = 0$$

where σ_{θ} , σ_z , and σ_r are the circumferential (hoop), axial, and radial stresses, respectively; p is the difference between internal and external pressures; d_m is the midwall tube diameter; and w is the tube wall thickness. Effective stress is given by:

$$\begin{aligned}\sigma_e &= \frac{1}{\sqrt{2}} [(\sigma_{\theta} - \sigma_z)^2 + (\sigma_z - \sigma_r)^2 + (\sigma_r - \sigma_{\theta})^2]^{1/2} \\ &= \frac{\sqrt{3}}{2} \sigma_{\theta}\end{aligned}$$

This computation of effective stress is in good agreement with the midwall effective stress computed from exact elastic solutions for a pressurized tube. We expect midwall stresses to change very little as a result of stress redistribution accompanying creep deformation. Consequently, we elected to describe deformation in terms of midwall hoop strain, since the selection of hoop strain at the external or internal diameter would result in a greater distortion of the correct effective stress-effective strain relation. Measured hoop strains at the external diameter were converted to midwall hoop strain based on conservation of volume with zero axial strain; for the present specimen geometry, measured strains were multiplied by 1.15 to obtain midwall hoop strains.

Specimens employed in this study were pressurized and sealed, so assuming uniform deformation, the pressure-volume product is constant as the specimen deforms. As a result, the stress does not increase with strain as it does in a specimen subjected to a constant pressure, but remains nearly constant.

2. Results

Experimental hoop strain values for various creep times and stresses are tabulated in Reference 22. Our analysis begins with a description of the relation between strain and stress with time as a parameter. This starting point contrasts with that frequently used in describing uniaxial creep results, which begins with a formulation of the time dependence of creep strain with stress as a parameter. The basic reason for this difference is the widely spaced time intervals between strain measurements in the present experiment, but it is interesting to note that early attempts to begin the analysis with a strain-time relation failed to identify an analytical form which would apply over the complete time and temperature ranges. As we shall see, however, a consistent description of time dependence is obtained when we begin with the strain-stress relation.

The experimental results consistently show a linear dependence of strain on stress at low stress levels, with a much stronger stress dependence appearing at higher stress levels.

As illustrated in Figure HEDL.34, we described this behavior as the sum of two strain components, one which exhibits a linear stress dependence and one which exhibits some stronger stress dependence. In evaluating the slope, $C(t)$, of the linear strain-stress relation, the fit to the experimental data was allowed to intercept the strain axis at non-zero values (Figure HEDL.34). These strain intercept values, ϵ_0 , were generally in the range $\pm 0.02\%$, which is considered to be within the measurement uncertainty, and showed no systematic variation with either time or temperature. We then define creep strain, ϵ_c , as:

$$\epsilon_c = \epsilon - \epsilon_0$$

where ϵ is the measured hoop strain and ϵ_0 is the intercept strain which may be either positive or negative. Our description of the two strain components is then given by:

$$\epsilon_1 = C(t) \sigma$$

$$\epsilon_2 = \epsilon_c - \epsilon_1 = \epsilon_c - C(t) \sigma.$$

Values of $C(t)$ were determined from each set of strain-stress data, and are plotted against $\log(t)$ in Figure HEDL.35. These results were described by an equation of the form:

$$C(t) = A \cosh^{-1}(1+rt)$$

where $\cosh^{-1} x = \ln(x + \sqrt{x^2 + 1})$, as shown in Figure HEDL.35. Swindeman and Pugh of ORNL suggested the inverse hyperbolic cosine function for describing creep data, but did not explore its application in detail. This function has the characteristics of $\ln(1+rt)$ at long times, but does not decrease as rapidly as $\ln(1+rt)$ at short times. Blackburn et al. (24) reported results of a uniaxial creep test on a rod specimen at conditions within the present linear stress dependence range (i.e., 850°F, 20,000 psi). These data are plotted in Figure HEDL.36, along with analytical descriptions based on both $\cosh^{-1}(1+rt)$ and $\ln(1+rt)$. The $\cosh^{-1}(1+rt)$ function clearly yields the better description of the uniaxial experiment results, and was therefore selected for describing the present $C(t)$ results. Values of the parameters A and r derived from the $C(t)$ results in Figure HEDL.36 are presented in Figure HEDL.37, along with the analytical description of the temperature dependence of these parameters. The complete equations describing the strain component with linear stress dependence are as follows:

$$\epsilon_1 = A_\sigma \cosh^{-1}(1+rt)$$

$$\ln A = \begin{cases} -8.94451 - \frac{4331.4}{T} \\ \text{or} \\ -1.3748 - \frac{10747.1}{T} \end{cases}$$

whichever yields the smaller value of $\ln A$,

$$\ln r = \begin{cases} 291.069 - \frac{926425.0}{T} + \frac{1.0114 \times 10^9}{T^2} - \frac{3.70757 \times 10^{11}}{T^3} \\ \text{or} \\ -2.99573 \end{cases}$$

whichever yields the larger value of $\ln r$,

FIGURE HEDL.34. Illustration of Analysis Procedure for Strain-Stress Data.

FIGURE HEDL.35. Experimental Results and Analytical Description for $C(t)$.

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FIGURE HEDL.36. Analytical Descriptions of Creep Data Using $\cosh^{-1} (1+rt)$ and $\ln (1+rt)$ Functions.

HEDL 7412-22.6

FIGURE HEDL.37. Temperature Dependence of A and r in the Relation $\epsilon^C = A\sigma \cosh^{-1}(1 + rt)$.

where T is temperature in °K ($T = \frac{5(F-32)}{9} + 273.15$, where F is temperature in °F).

We turn now to a description of the strain component ϵ_2 , which exhibits a strong stress dependence. We represent this strain component with an equation of the form:

$$\epsilon_2 = K(t) \sigma^n$$

Figure HEDL.38 shows a plot of $\log \epsilon_2$ against $\log \sigma$ for the 2410-hour data illustrating this relation. We obtain an n value of 6.3 at temperatures of 1200°F and below and a value of 4.4 at 1400°F. The results in Figure HEDL.38 for the three lower temperatures yield a well defined n value which represents the data well over the whole time range. The n value at 1400°F is not as well defined by the results in Figure HEDL.38, and the selected value of 4.4 was judged to give the best overall representation of all the 1400°F data. The temperature dependence of n is described by the following equations:.

For $T \leq 922.039^\circ\text{K}$ (1200°F)

$$n = 6.3$$

For $T > 922.039^\circ\text{K}$ (1200°F)

$$n = -124.539 + 0.283804 T - 1.539 \times 10^{-4} T^2$$

The polynomial coefficients were evaluated by requiring that $n = 6.3$ at 1200°F, $n = 4.4$ at 1400°F, and $\frac{dn}{dT} = 0$ at 1200°F, thus providing a smooth transition from the constant n value at lower temperatures.

Values of $K(t)$ at each time where nonlinear stress dependence was observed were obtained from a series of plots similar to Figure HEDL.38. The variation of $K(t)$ with time is illustrated in Figure HEDL.39. At short times, $K(t)$ can be represented by the relation:

$$K(t) = K_1 t^m$$

where m is 0.533 at 1200°F and below, but is 1.172 at 1400°F. The temperature dependence of m is given by the equations:

For $T \leq 922.039^\circ\text{K}$ (1200°F)

$$m = 0.533$$

For $T \geq 922.039^\circ\text{K}$ (1200°F)

$$m = 44.5365 - 0.954482 T + 5.17593 \times 10^{-5} T^2$$

Polynomial coefficients were evaluated by requiring that $m = 0.533$ at 1200°F, $m = 1.172$ at 1400°F, and $\frac{dm}{dT} = 0$ at 1200°F, thus assuring a smooth transition from the constant m value at lower temperatures. There is clearly a departure from the $K_1 t^m$ relation at longer times in the direction of a stronger time dependence for the results at 1000, 1200 and 1400°F. This behavior was described by adding another term, $K_2 t^{2.5}$, to the above relation, as illustrated in Figure HEDL.39. The equation describing $K(t)$ becomes:

$$K(t) = K_1 t^m + K_2 t^{2.5}$$

Values of K_1 and K_2 obtained from Figure HEDL.39 are presented in Figure HEDL.40, where the temperature dependence of these parameters is given by the equations:

HEDL 7412-22.4

FIGURE HEDL.38. Illustration of the Stress Dependence of Strain Component ϵ_2 .

HEDL 7412-22.5

FIGURE HEDL.39. Time Dependence of $K(t)$ in the Relation $\epsilon_2 = K(t)\sigma^n$.

FIGURE HEDL.40. Temperature Dependence of K_1 and K_2 in the Relation $\epsilon_2 = K_1 \sigma^n t^m + K_2 \sigma^n t^{2.5}$.

For 1033.15°K (1400°F) $\geq T \geq 810.928^{\circ}\text{K}$ (1000°F)

$$\ln K_1 = 5131.26 - \frac{1.35198 \times 10^7}{T} + \frac{1.17285 \times 10^{10}}{T^2} - \frac{3.39674 \times 10^{12}}{T^3}$$

For $T < 810.928^{\circ}\text{K}$ (1000°F)

$$\ln K_1 = 35.3606 - \frac{89658.6}{T}$$

For 1033.15°K (1400°F) $\geq T \geq 866.483^{\circ}\text{K}$ (1100°F)

$$\ln K_2 = 1179.06 - \frac{2.19791 \times 10^6}{T} + \frac{9.52226 \times 10^8}{T^2}$$

For 866.483°K (1000°F) $> T \geq 810.928^{\circ}\text{K}$ (1000°F)

$$\ln K_2 = -89.2335$$

For $T < 810.928^{\circ}\text{K}$ (1000°F)

$$\ln K_2 = -453.917 + \frac{5.91409 \times 10^5}{T} - \frac{2.39794 \times 10^8}{T^2}$$

The extrapolation of $\ln K_1$ below 1000°F is made using the slope of the third order polynomial for the high temperature range evaluated at 1000°F . This assures a smooth transition in the temperature dependence of K_1 . Extrapolation of $\ln K_2$ below 1000°F is based on a second order polynomial in $1/T$, with coefficients evaluated to give $\ln K_2 = -89.2335$ at 1000°F , $d \ln K_2 / d(1/T) = 0$ at 1000°F , and $\ln K_2 = -96$ at 825°F . This again assures a smooth transition in the temperature dependence of K_2 , and the value of $\ln K_2$ imposed at 825°F assures that the strain contribution from the term containing K_2 is negligible at this low temperature. We impose this latter requirement since the experimental hoop strain data at 825°F show no evidence of nonlinear stress dependence (see Figure HEDL.49).

Our final description of creep strain is therefore:

$$\epsilon^C = A \sigma \cosh^{-1} (1+rt) + K_1 \sigma^n t^m + K_2 \sigma^n t^{2.5}.$$

Using the numerical values of the parameters A , r , K_1 , K_2 , n and m obtained from the equations given earlier, this relation predicts the midwall hoop strain, ϵ^C , in percent when hoop stress is given in psi (using thin-wall approximation), and time, t , is given in hours.

4. Evaluation of Results

The description of creep behavior given by the equations derived in the preceding section is compared with the experimental data in Figures HEDL.41 through .49. Comparisons in Figures HEDL.41 through .44 are shown on $\log \epsilon^C$ against $\log t$ plots, with experimental points plotted as the measured strain minus ϵ_0 (see Figure HEDL.34). In general, the analytical description of the experimental results is good at 1400°F (Figure HEDL.41), although the equations overpredict creep strain for longer times at 5.4 ksi and underpredict strain at long times at 1.5 ksi. Specimen failure is observed at the three highest stresses as evidenced by a constant value of creep strain for the longer times. At 1200 and 1100°F , the creep equations again provide a good description of the experimental data (Figures HEDL.42

FIGURE HEDL.41. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1400°F.

FIGURE HEDL.42. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1200°F.

FIGURE HEDL.43. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1100°F. HEDL 7412-22.10

FIGURE HEDL.44. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1000°F. HEDL 7412-22.11

HEDL 7412-22.12

FIGURE HEDL.45. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1400°F.

HEDL 7412-22.13

FIGURE HEDL.46. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1200°F.

FIGURE HEDL.47. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1100°F.

FIGURE HEDL.48. Comparison of Analytical Creep Equation Predictions with Experimental Data at 1000°F.

HEDL 7412-22.24

FIGURE HEDL.49. Comparison of Analytical Creep Equation Predictions with Experimental Data at 825°F.

and .43). The comparison for 1000°F shown in Figure HEDL.44 shows substantial scatter in the experimental results. We estimate the experimental uncertainty for our strain measurements to be in the range ± 0.02 to $\pm 0.04\%$, so the scatter appears very large in logarithmic coordinates at these low strain values. However, when the comparison is viewed in linear coordinates (see Figure HEDL.48), the description appears quite reasonable.

Figures HEDL.45 through .48 present comparisons of creep equations and experimental data in linear strain-time coordinates. The computed creep curves show the typical features of creep behavior: an initial period of primary creep, a minimum creep rate, and then accelerating creep in third stage. A number of the low stress curves appear to exhibit a steady-state creep rate, even though a steady-state creep term is not included in the analytical equations.

Experimental data at 825°F did not give a clear picture of creep behavior because the small strains were near the limit of detectability. However, the results shown in Figure HEDL.49 indicate that the procedure of deriving the creep equation from the uniaxial data at 850°F and suppressing strain contribution from terms containing K_1 and K_2 yields a very reasonable representation of the data. (Note: Strain values plotted in Figure HEDL.49 were not adjusted by subtracting ϵ_0 .)

Lovell⁽²⁵⁾ formulated creep equations for 20% cold-worked Type 316 stainless steel based on results from uniaxial creep tests, but these equations tend to overpredict the present pressurized tube results.⁽²²⁾ We have made a detailed comparison of the data reported by Lovell for tube and rod specimens with the present experimental results on pressurized tubes.* One illustration of these comparisons is shown in the isochronous effective strain-effective stress plot of Figure HEDL.50. (Note: Plotted strain values for biaxial tests were not adjusted by subtracting ϵ_0 .) Although the scatter is larger in the uniaxial results, we find basically good agreement between the uniaxial and biaxial results at low stresses, particularly for the tube material. However, at high stresses larger strains are observed in uniaxial tests than are predicted from extrapolation of the biaxial data. These observations were quite general for the temperature range from 1000 to 1400°F for times to 2400 hours. Since the pressurized tube data give a more complete definition of the low stress range, we prefer the present creep equations for describing both uniaxial and biaxial results.

The present creep equations may find application as input to constitutive equations for inelastic analysis of reactor components. Most computer programs require the materials creep description in terms of creep strain rate rather than creep strain for such calculations. We have therefore examined the behavior of the time derivative of present creep equations. Figures HEDL.51 through .53 show the relation between creep rate and creep strain at 1000, 1200 and 1400°F. The relation is smooth and continuous, as it should be for application in inelastic analyses. We also found that the creep rate was a smooth, continuous function of temperature.

One problem for the application of the current creep equation in inelastic analyses is the definition of creep rate at $t = 0$. The equation for creep rate is:

*The tube material is identical in the two studies and the rod material was fabricated from the same heat of steel.

HEDL-56

HEDL 7412-22.23

FIGURE HEDL.50. Comparison of 100-Hour Creep Data at 1200°F for Uniaxial and Biaxial Loading.

HEDL 7412-22.18

FIGURE HEDL.51. Computed Relation Between Creep Rate and Creep Strain at 1000°F.

HEDL-57

HEDL 7412-22.17

FIGURE HEDL.52. Computed Relation Between Creep Rate and Creep Strain at 1200°F.

HEDL 7412-22.16

FIGURE HEDL.53. Computed Relation Between Creep Rate and Creep Strain at 1400°F.

$$\frac{d\epsilon^c}{dt} = \frac{A\sigma r}{\sqrt{2rt + r^2t^2}} + mK_1\sigma^n t^{m-1} + 2.5K_2\sigma^n t^{1.5}$$

If $m < 1$, both the first and second terms give infinite creep rates at $t = 0$. To circumvent this singularity we propose specifying a finite, constant creep rate over some short time interval from $t = 0$ to $t = t_0$. The creep rate beyond t_0 is then given by the time derivative of the creep equation. We examined this procedure to select a value of t_0 which would not significantly alter the agreement between the creep equation and the experimental data. We arrived at the following procedure for using the creep equations in inelastic analysis:

$$\frac{d\epsilon^c}{dt} = \frac{d\epsilon_1^c}{dt} + \frac{d\epsilon_2^c}{dt} + \frac{d\epsilon_3^c}{dt}$$

For $t = 0$

$$\frac{d\epsilon_1^c}{dt} = \frac{A\sigma r}{\sqrt{0.002 r + 1 \times 10^{-6} r^2}}$$

$$\frac{d\epsilon_2^c}{dt} = \begin{cases} mK_1\sigma^n (0.001)^{m-1} & \text{for } m < 1 \\ K_1\sigma^n & \text{for } m = 1 \\ 0 & \text{for } m > 1 \end{cases}$$

$$\frac{d\epsilon_3^c}{dt} = 0$$

For $t > 0$

$$\frac{d\epsilon_1^c}{dt} = \begin{cases} \frac{A\sigma r}{\sqrt{2rt + r^2t^2}} \\ \text{or} \\ \frac{A\sigma r}{\sqrt{0.002 r + 1 \times 10^{-6} r^2}} \end{cases}$$

whichever yields the smaller value of $\frac{d\epsilon_1^c}{dt}$

$$\frac{d\epsilon_2^c}{dt} = \begin{cases} mK_1\sigma^n t^{m-1} \\ \text{or} \\ mK_1\sigma^n (0.001)^{m-1} \end{cases} \quad [\text{applies only if } m < 1]$$

whichever yields the smaller value of $\frac{d\epsilon_2^c}{dt}$

$$\frac{d\epsilon_3^c}{dt} = 2.5 K_2\sigma^n t^{1.5}$$

The corresponding equations for strain are:

$$\epsilon^c = \epsilon_1^c + \epsilon_2^c + \epsilon_3^c$$

For $t \leq 0.001$ hours

$$\epsilon_1^c = \frac{A\sigma r t}{\sqrt{0.002 r + 1 \times 10^{-6} r^2}}$$

$$\epsilon_2^c = \begin{cases} m K_1 \sigma^n (0.001)^{m-1} t & \text{for } m < 1 \\ K_1 \sigma^n t^m & \text{for } m \geq 1 \end{cases}$$

$$\epsilon_3^c = K_2 \sigma^n t^{2.5}$$

For $t > 0.001$ hours

$$\epsilon_1^c = A\sigma \cosh^{-1}(1+rt) + \frac{0.001 A\sigma r}{\sqrt{0.002 r + 1 \times 10^{-6} r^2}} - A\sigma \cosh^{-1}(1+0.001 r)$$

$$\epsilon_2^c = \begin{cases} K_1 \sigma^n t^m + m K_1 \sigma^n (0.001)^m - K_1 \sigma^n (0.001)^m & \text{if } m < 1 \\ K_1 \sigma^n t^m & \text{if } m \geq 1 \end{cases}$$

$$\epsilon_3^c = K_2 \sigma^n t^{2.5}$$

To convert these equations to effective strain, $\bar{\epsilon}$, and effective stress, $\bar{\sigma}$, units, replace ϵ by $\sqrt{3/2} \bar{\epsilon}$ and σ by $2/\sqrt{3} \bar{\sigma}$.

5. Conclusions and/or Recommendations

The analytical equations developed here are a good representation of the thermal creep deformation of 20% cold-worked Type 316 stainless steel over the temperature range from 825 to 1400°F for times to 2400 hours at stresses from zero to as high as 60,000 psi.

6. Expected Achievements

Experimental tests are still in progress, and 4000-hour creep data should be obtained during the next quarter.

E. IRRADIATION CREEP IN COLD-WORKED TYPE 316 STAINLESS STEEL

E. R. Gilbert, J. L. Straalsund, J. J. Holmes

1. Objective

The objective of this study is to determine the irradiation creep behavior of 20% cold-worked Type 316 stainless steel over the range of exposure conditions relevant to LMFBRs.

2. Accomplishments and Status

a. Scope of Work. The X-157 Stress Effects Experiment consists of 1-inch long segments of pressurized N-2 lot tubing. Forty-three 20% cold-worked specimens per fluence level have been measured after irradiation to peak fluences of 0.4, 1.6 and 3.0×10^{22} n/cm² ($E > 0.1$ MeV). Specimens now being irradiated are scheduled to be measured February 1976 after irradiation to peak fluence levels of 6.0 and 10.0×10^{22} n/cm² ($E > 0.1$ MeV). Hoop stresses range from 0 to 60,000 psi with irradiation temperatures in the range of 710 to 1070°F.

The methods of specimen manufacture, pressure application, and measurements were the same as used for the thermal control specimens described in Reference 26.

b. Results. A listing of the data is presented in Table HEDL.9. The hoop stress was computed by the thin wall formula for the tangential stress at the midwall and is 7.167 times the pressure differential across the tube wall. An elastic solution estimate of the midwall hoop strain was used as the strain variable. This was obtained by multiplying the fractional change in the outer diameter ($\Delta D/D_0$) by a factor of 1.15.

The diameter measurements for low stress specimens indicate negative strains. Length measurements also indicate negative axial strains. These axial strains are independent of stress. These reductions in diameter and length are comparable to values of thermal densification reported by Straalsund and Paxton for stainless steel.⁽²⁷⁾

c. Evaluation of Results. The first step in the data analysis involved constructing isochronal strain-stress curves for each temperature and each fluence set. These curves appeared linear in hoop stress and showed negative strain intercepts at zero stress. A least squares analysis of the data points yielded a positive slope $d\epsilon_h/d\sigma_h)_{G_D} \phi t$ and an intercept b which was negative. Since the corresponding b_a for axial strain is independent of stress, it was assumed that the diametral b is also independent of stress. Consequently, the strain values at all stresses were increased by the absolute value of b so that the creep data would show zero strain at zero stress. Datum points for 930°F after this adjustment are shown in Figure HEDL.54. The specific values of $d\epsilon_h/d\sigma_h)_{G_D} \phi t$ and b for each data set are given in Table HEDL.10.

The least square values of $d\epsilon_h/d\sigma_h)_{G_D} \phi t$ were then plotted versus ϕt as shown in Figure HEDL.55. At temperatures below 965°F the datum points are linear with fluence. A least squares analysis of the datum points yielded values for the transient and steady-state irradiation creep coefficients A_h and B_h for the following equation -

$$\epsilon_h = A_h \sigma_h + B_h \sigma_h \phi t \quad (1)$$

Values for these coefficients are given in Table HEDL.11 for atom displacement according to the Doran damage function, for fast fluence, and for $\bar{E} \phi t$. For temperatures of 710, 845 and 930°F the values of A_h appear to scatter closely around zero, while the value of B_h is $8.6 \pm 2.0 \times 10^{-9} \text{ psi}^{-1} \text{ dpa}^{-1}$. For temperatures above 930°F, the data show systematic deviations from Equation 1. The creep rate increases with fluence so that the linear analysis results is a negative value of A_h and an average value of B_h . Since there is no swelling as confirmed by immersion density measurements, the deviation cannot be attributed to effects such as swelling-enhanced creep.⁽²⁸⁾

It is anticipated that the departure from linear fluence behavior is a consequence of irradiation induced microstructural changes in dislocation density and configuration. At the higher irradiation temperatures in this experiment, the Frank loops may readily unfault and become mobile dislocations. The cold-worked microstructure also slowly recovers during irradiation at these higher temperatures and the rearrangement of the dislocations results in accelerated creep. It is also possible that irradiation enhanced recovery at the higher irradiation temperatures could cause the thermal creep rate to increase with time. One complication in the analysis of these data is the feature that the specimens were given a

TABLE HEDL.9
IN-REACTOR CREEP DATA -- X157

HOOP ⁽¹⁾ STRESS (ksi)	IRRAD. TEMP. (°F)	X157 1553 MWD						X157A 6192 MWD			X157B 10775 MWD		
		R (cm)	Z (cm)	$\Delta D/D_0$ (10 ⁻⁶)	$G_{D\dot{t}}$ (dpa)	$(10^{22}\dot{t} \text{ n/cm}^2)$ (E>0.1 MeV)		$\Delta D/D_0$ (10 ⁻⁶)	$G_{D\dot{t}}$ (dpa)	$(10^{22}\dot{t} \text{ n/cm}^2)$ (E>0.1 MeV)	$\Delta D/D_0$ (10 ⁻⁶)	$G_{D\dot{t}}$ (dpa)	$(10^{22}\dot{t} \text{ n/cm}^2)$ (E>0.1 MeV)
0	710	17.7	-30.0	-163	.48	.126		-388	1.91	.502	-329	3.33	.874
5	710	17.7	-30.0	-148	.48	.126		-203	1.91	.502	62	3.33	.874
10	710	15.1	-30.0	-109	.49	.128		-91	1.95	.510	145	3.40	.890
20	710	15.1	-30.0	-7	.49	.128		-254	1.95	.510			
30	710	15.1	-30.0	43	.49	.128		11	1.95	.510	406	3.40	.890
40	710	17.7	-30.0	-11	.48	.126		116	1.91	.502	496	3.33	.874
60	710	14.3	-30.0	181	.49	.126		413	1.95	.502	732	3.40	.874
0	845	14.3	27.2	-134	.59	.150		-98	2.35	.598	69	4.1	1.04
4.9	800	14.3	-23.6	-178	.87	.214		127	3.47	.853	428	6.04	1.48
7.8	845	15.1	27.2	-65	.59	.152		130	2.35	.606	58	4.1	1.05
10.3	845	17.7	27.2	-43	.58	.151		15	2.31	.602	83	4.02	1.05
15.4	845	17.7	27.2	-22	.58	.151		58	2.31	.602	373	4.02	1.05
20.6	845	15.1	27.2	94	.59	.152		305	2.35	.606	526	4.1	1.05
25.8	845	15.1	27.2	-51	.59	.152		453	2.35	.606	713	4.1	1.05
30.9	845	17.7	27.2	120	.58	.152		257	2.31	.606	831	4.02	1.05
34.1	800	14.3	-23.6	221	.87	.214		648	3.47	.853	1780	6.04	1.48
41.2	845	14.3	27.2	54	.59	.150		558	2.35	.598	1399	4.1	1.04
58.5	800	17.7	-17.3	623	1.4	.310		1925	5.6	1.24	3946	9.7	2.15
0	930	17.7	8.1	-213	1.8	.369		-330	7.2	1.47	-170	12.5	2.56
2.4	930	15.1	14.5	-65	1.44	.286		-203	5.7	1.14	275	10.0	1.98
4.8	930	17.7	14.5	-141	1.38	.284		-152	5.5	1.13	-18	9.57	1.97
7.2	930	15.1	14.5	15	1.44	.286		-214	5.7	1.14	496	10.0	1.98
9.6	930	14.3	14.5	-3	1.47	.296		232	5.86	1.18			
14.3	930	14.3	14.5	112	1.47	.296		457	5.86	1.18	1020	10.2	2.05
18.8	930	17.7	8.1	163	1.8	.369		598	7.2	1.47	1601	12.5	2.56
23.9	930	14.3	14.5	213	1.47	.296		797	5.86	1.18	2018	10.2	2.05
28.3	930	17.7	8.1	340	1.8	.369		1125	7.2	1.47	2192	12.5	2.56
38.2	930	15.1	14.5	459	1.44	.286		1408	5.7	1.14	2971	10.0	1.98
47.1	930	15.1	8.1	---	---	---		---	---	---	4830	13.7	2.76
56.4	930	15.1	8.1	807	1.97	.398		3040	7.85	1.59	---	---	---
0	955	14.3	-17.3	-171	1.71	.33		-178	6.82	1.32	-4	11.9	2.29
4.8	985	15.1	-23.6	-72	.87	.210		-120	3.47	.84	105	6.04	1.46
9.6	1035	15.1	20.8	-40	.89	.206		-29	3.55	.82	361	6.17	1.43
19.1	1035	14.3	20.8	83	.91	.206		442	3.63	.82	1297	6.31	1.43
33.3	1035	17.7	20.8	---	---	---		---	---	---	1960	6.11	1.43
38.1	1035	17.7	20.8	264	.88	.206		1127	3.51	.82	---	---	---
0	965	17.7	-10.9	-109	1.89	.380		-500	7.54	1.52	-431	13.1	2.64
2.1	965	14.3	-4.6	-40	2.17	.434		-127	8.65	1.73	315	15.1	3.01
4.3	965	15.1	-10.9	-54	1.98	.392		14	7.89	1.56	344	13.7	2.72
8.6	965	15.1	-4.6	-40	2.10	.421		-76	8.37	1.68	717	14.6	2.92
21.4	965	14.3	-10.9	---	---	---		---	---	---	2730	14.2	2.80
25.7	965	14.3	-10.9	---	---	---		1167	8.13	1.61	---	---	---
29.9	965	14.3	-10.9	145	2.04	.403		---	---	---	---	---	---
0	1070	17.7	1.8	-134	1.97	.400		-326	7.85	1.59	-244	13.7	2.78
0.82	1070	17.7	-4.6	-123	2.00	.406		-293	7.97	1.62	-102	13.9	2.82
2.1	1070	15.1	1.8	-217	2.05	.414		-420	8.17	1.65	157	14.2	2.87
6.2	1070	14.3	1.8	---	---	---		---	---	---	1229	14.9	2.98
8.2	1070	14.3	1.8	---	---	---		496	8.57	1.71	---	---	---
12.2	1070	14.3	1.8	58	2.15	.429		---	---	---	---	---	---

(1) Hoop Stress = $\frac{P D_{midwall}}{2t} = 7.167 P$.

FIGURE HEDL.54. In-Reactor Creep X-157 (930°F).

FIGURE HEDL.55. In Reactor Creep X-157 (710 to 1070°F).

TABLE HEDL.10

VALUES OF $d\epsilon_h/d\sigma_h)_{DPA}$ AND b FOR X157 PRESSURIZED TUBE EXPERIMENT

Irradiation Temperature, °F	EBR-II Exposure MWD	(1) (2) $d\epsilon_h/d\sigma_h)_{DPA}$	b 10^{-6}	Coefficient or Determination	Average $G_D \phi t$, DPA
		10^{-9} psi^{-1}			
710	1,553	6.2	-182	.90	.49
710	6,192	13.5	-383	.88	1.93
710	10,775	17.4	-131	.89	3.37
845	1,553	5.6	-114	.57	.59
845	6,192	17.4	-90	.82	2.32
845	10,775	38.9	-158	.94	4.05
930	1,553	19.2	-182	.96	1.55
930	6,192	55.6	-392	.97	6.18
930	10,775	96	-145	.98	10.76
1035	1,553	11.8	-145	.995	.89
1035	6,192	44.4	-385	.993	3.54
1035	10,775	77.2	-217	.97	6.16
965	1,553	8.9	-102	.95	2.02
965	6,192	68.2	-444	.94	8.12
965	10,775	158	-307	.97	14.14
1070	1,553	20.4	-197	.76	2.05
1070	6,192	124	-500	.89	8.17
1070	10,775	280	-335	.995	14.2

$$(1) \epsilon_h = 1.15 \Delta D/D_0$$

$$(2) \sigma_h = \frac{P D_{\text{midwall}}}{2t} = 7.167 P$$

TABLE HEDL.11

CREEP COEFFICIENTS A_h AND B_h FOR X157 20% COLD-WORKED 316 SS PRESSURIZED TUBES

Irradiation Temperature, °F	A_h	B_h	B_h	B_h
	10^{-9} psi^{-1}	$10^{-9} \text{ psi}^{-1} \text{ dpa}^{-1}$	$10^{-30} \text{ psi}^{-1} \text{ cm}^2 \text{ n}^{-1}$ ($E > 0.1 \text{ MeV}$)	$10^{-30} (\text{psi MeV n/cm}^2)^{-1}$ ($E > 10^{-10} \text{ MeV}$)
710	3.2	6.6	3.1	4.4
845	-4.0	10.7	5.1	6.7
930	6.9	8.5	4.0	5.0
1035	1.6	12.0	5.8	7.3
965	-24.5	15	7.2	9.1
1070	-29.6	19.8	9.4	11.0

thermal set prior to irradiation which was different from the attained irradiation temperatures. The thermal set treatment and the irradiation temperatures are given in Table HEDL.12. These short thermal set treatments may have resulted in some recovery of the cold-work and some aging thus causing more thermal creep than observed in 20% cold-worked Type 316 stainless steel tubing. However, recent strain data obtained from Bloom at Oak Ridge National Laboratory on irradiated pressurized 20% cold-worked Type 316 stainless steel tubes which were not given any thermal treatment prior to irradiation show similar strains indicating that the thermal set treatment did not have an appreciable influence on the creep strain. Further thermal control experiments are planned which will help resolve these questions.

TABLE HEDL.12
DESCRIPTION OF THE THERMAL SET TREATMENT

<u>Irradiation Temperature (°F)</u>	<u>Thermal Set Temperature (°F)</u>	<u>Thermal Set Time (Hours)</u>
1070	1400	1
1035	1100	1
965	1200	1
930	1000	2
845	825	4
710	705	4

The increase in creep rate with neutron fluence is described with a hyperbolic tangent function by the curves drawn in Figure HEDL.55. The equation is given below.

$$\frac{\epsilon_h}{\sigma_h} = R_1 [P - (R_1/15) \tanh (10 P/r_1)] \times 10^{-8} \quad (2)$$

$$\epsilon_h = \text{midwall hoop strain} = 1.15 \Delta D/D_0$$

$$\sigma_h = \text{midwall hoop strain, psi} = 7.167 P$$

$$R_1 = 2.3 + (16.9 \times 10^6) \exp (-24,000/RT)$$

$$P = \text{neutron fluence, } 10^{22} \text{ n/cm}^2 \text{ (E > 0.1 MeV)}$$

$$T = \text{temperature, } ^\circ\text{K}$$

Thermal control creep tests were conducted on the same material which was used for the in-reactor experiment.⁽²⁹⁾ The thermal creep strains were subtracted from the in-reactor and plotted in Figure HEDL.56. The resulting strain obtained at the different temperatures appears to form a narrow band up to $1 \times 10^{22} \text{ n/cm}^2$. A hyperbolic tangent function is plotted in Figure HEDL.56 which describes the difference between the total in-reactor strain and the thermal creep strain measured in control experiments. This equation is:

$$\frac{\epsilon_h}{\sigma_h} = \{2.3P + A [P-1.7 \tanh (P/1.7)]\} \times 10^{-8}$$

$$A = 25,000 \exp (-13,000/RT) \quad (3)$$

The other terms retain the previously assigned definitions.

3. Conclusions and/or Recommendations

These results show that in-reactor creep at temperatures approaching 1100°F exceeds thermal creep. This stress dependence is predominantly linear for fluences up to 3×10^{22} n/cm² (E > 0.1 MeV), but the strains increase exponentially both with fluence and temperature. Existing creep correlations do not predict this behavior and underpredict the strains at temperatures exceeding 800°F.

4. Applications

For neutron fluences greater than 1×10^{22} n/cm² the values of $d\epsilon_h/d\sigma_h$ follow Equations 1, 2 and 3 to approximately $\pm 25\%$. Equations (1) and (2) should be used to predict total in-reactor creep strains. Equation (3) should be used in addition to the thermal creep equation⁽²⁹⁾ strain as shown in Figure HEDL.57. Equation (2) is expected to provide better extrapolation to higher fluences than Equation (1) and therefore may be preferred over Equation (1).

5. Expected Achievements

Two sets of pressurized tubes are continuing in EBR-II to goal fluences of 6 and 10^{22} n/cm² (E > 0.1 MeV). These specimens are expected to be available early in 1976.

6. References

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FIGURE HEDL.56. X-157 In-Reactor Creep Less Thermal Creep.

FIGURE HEDL.57. Comparison of Strain in Pressurized Tubes (X-157) with Combined Thermal and Irradiation Creep Equations.

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II. MATERIALS ANALYSIS AND APPLICATION

H. H. Yoshikawa

A. FULL POWER DOSIMETRY EXPERIMENT IN THE EBR-II

J. A. Ulseth, E. P. Lippincott, B. L. Combs, and W. N. McElroy

1. Objective

The primary objective of this experiment is to provide more complete and accurate neutron flux and neutron energy descriptions in specific regions of EBR-II where fuel, absorber, and structural material irradiations are performed.

A second objective of the full power dosimetry test is to provide timely data on the feasibility of utilizing FTR wire wrap directly as a dosimeter material for fuel irradiations.

This experiment will provide data to permit an assessment of the agreement of results from present FTR calculational procedures with dosimetry measurements and results from ANL calculational techniques.

2. Summary

Six B-19 subassemblies located at 2D1, 4C3, 4E1, 7F4, 8F4 and 8F6 for the Full Power Dosimetry Experiment were irradiated for eight full power days in the EBR-II during Run 75D for a total of 582 MWD. All the very short half-life monitors were counted by January 25, 1975. Completed radiometric results on the $^{63}\text{Cu}(n,\gamma)^{64}\text{Cu}$ were made available for analysis on January 29, 1975.

3. Accomplishments and Status

The replacement of the radial depleted uranium blanket with a stainless steel reflector in Rows 7 and 8 after Run 56A has appreciably altered the nuclear environment of the core and reflector regions of the EBR-II. Driver fuel and experimental fuel subassemblies occupy approximately one-half of the Row 7 subassembly positions, and will occupy a number of Row 8 positions. In addition, high worth control rods have been installed in Row 5.

The full power dosimetry experiments conducted during Runs 31F⁽¹⁾ and 50H⁽²⁾ provided a means of predicting irradiation conditions for experiments that were being planned during the Run 31F and 50H time periods up to Run 56A. In addition, neutron environment characterization for experiments could be reasonably approximated from the flux spectra maps generated from the reaction data of Runs 31F and 50H. This dosimetry experiment is intended to provide flux spectra characterization for HEDL experiments that have been irradiated since Run 56A.

Both ANL-Chicago and Atomics International participated in this experiment. ANL irradiated one container of candidate CTR dosimetry materials and AI included five containers of He production monitors.

The AI containers include both bare and Au encapsulated candidate fluence monitors to determine if and to what extent the irradiation produced helium escapes from the monitor. Also FTR reference clad constituents were irradiated to determine helium production rates.

FTR wire wrap, FTR oxide fuel material, ^{240}Pu , ^{241}Pu and boron monitors are included to provide a basis for developing data for determining burnup, fission rates, fission gas

generation rates and boron absorber capture rates of FTR components, fuels, and control rods.

The dosimetry experiment subassemblies at 2D1, 4C3, 4E1, 7F4, 8F4 and 8F6 were irradiated during EBR-II reactor Run 75D for eight days at full power. The specific locations of the elements within the subassemblies, and the axial location of the monitors within the elements were described in the HEDL Technical Quarterly - Materials Technology report for October, November, and December 1974 (HEDL-TME 74-4). The types and numbers of monitors were also described in the above report. All monitors have been recovered from the experiment and all short half-life monitors were shipped to HEDL and radiometrically analyzed within six days after reactor shutdown. Counting of long half-life monitors has been started.

4. Expected Achievements

By the next reporting period, the midplane flux-spectra distribution will be completed to show the effect of the stainless steel reflector on the EBR-II core environment as determined from multiple foil measurement data.

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B. HELIUM PRODUCTION CROSS-SECTIONS

W. N. McElroy, E. P. Lippincott, and H. Farrar IV

1. Objective

The objective of this task is to establish helium production cross-sections of adequate accuracy for the prediction of helium formation rates in neutron irradiation environments. Furthermore, the measurement of helium content of suitable materials is being developed as a means of higher accuracy fluence-spectra dosimetry for use in damage analysis.

2. Accomplishments and Current Status

Significant quantities of helium are generated in materials exposed to neutrons in typical LMFBR spectra. Therefore, it is important to have accurate cross-sections for helium producing reactions. Furthermore, the measurement of helium content of a material for which the energy dependent, helium producing cross-sections are known accurately should be an excellent means of fluence-spectra dosimetry for use in damage analysis.

Because of the inadequacy of evaluated energy dependent cross-sections of earlier versions of ENDF/B, a set of helium production curves for stainless steel and its constituents irradiated in LMFBR spectra was developed through a joint HEDL-Atomics International program.⁽¹⁾ Spectrum-averaged integral (n, α) cross-sections were measured by irradiating alloys, compounds, and pure elements in a variety of spectra, and measuring the resulting helium concentrations by high sensitivity mass spectrometry. The exposures were made in EBR-II during high power dosimetry tests which provided the necessary neutron flux-spectral data. Additional measurements for ^{10}B and ^6Li have been made in the Coupled Fast

Reactivity Measurements Facility (CFRMF). Details of this work are given elsewhere.⁽¹⁻⁴⁾ These helium production curves are in use to predict helium generation in stainless steel and its major constituents in fission reactor spectra for the LMFBR program.

A consistent set of evaluated energy dependent cross-sections giving reliable estimates of helium production in elemental materials for fission and fusion reactor dosimetry and damage analysis does not presently exist. The current status for fission reactor applications is shown in Table HEDL.13. This is a preliminary up-to-date tabulation of some important helium production cross-sections. ENDF/B-IV (n,α) cross-sections [including cross-sections for other helium production reactions such $(n,n'\alpha)$ where available], averaged over the fission neutron spectrum, are presented and compared with values tabulated by Birss.⁽⁵⁾ Substantial disagreements are apparent.

Preliminary values measured at Atomics International using helium gas mass spectrometry are tabulated in Column 4 of Table HEDL.13. The cross-sections are derived from specimens of pure elements irradiated in EBR-II during the Run 50H dosimetry test at a Row 4 location having a mean neutron energy of ~ 0.8 MeV. The cross-sections measured in EBR-II can be related directly to fission spectrum cross-sections because, at neutron energies sufficiently high for most (n,α) reactions to be significant, the EBR-II spectrum retains the fission spectrum shape. The measured helium production cross-sections from the Row 4 center position of EBR-II were therefore simply multiplied by a factor of 4.55 in order to present them in Column 4 of Table HEDL.13 as fission spectrum-averaged cross-sections.⁽¹⁾

The measured values in Column 4 are preliminary because several are based on only one or two measurements, although numerous additional irradiated specimens are presently awaiting analysis (not only from the Row 4 center location of EBR-II but also from other locations where the mean neutron energy is lower). In addition, the nitrogen impurity content of the specimens must be determined, because nitrogen has a significant helium generation rate in EBR-II neutron spectra. At present, the helium contribution from nitrogen (and perhaps boron) is thought to be low or negligible on the basis of a few nitrogen measurements and on the fact that, in most cases, similar helium concentrations are seen in specimens from different lots of a material.

The last column of Table HEDL.13 shows the ratio of the measured values to the ENDF/B-IV cross-sections. Ratios are generally greater than unity which could be attributed to one or more of the following:

- a. The calculations failed to take into account such reactions as $(n,n\alpha)$, $(n,2\alpha)$, etc. However, these reactions are expected to be negligible in fission reactors.
- b. The cross-section evaluations used in the calculations are inadequate, especially in the region of the effective thresholds.
- c. The measured values are in error due to errors in the total neutron fluence or in the factor used to convert spectrum-averaged to fission-averaged cross-sections. Both these numbers have uncertainties of a few percent. However, since all the pure elements were irradiated together at one location, such errors would not affect relative helium production rates but would bias the whole set.

TABLE HEDL.13

FISSION SPECTRUM AVERAGED HELIUM PRODUCTION CROSS-SECTIONS

ELEMENT OR ISOTOPE	Cross-Sections (mb)			MEAS./ CALC.
	Calc. Using ENDF/B-IV(a)	BIRSS (b)	MEASURED (c)	
⁶ Li	487. (d)	-	(e)	-
⁷ Li	22.9	25.8	(e)	-
¹⁰ B	515. (d)	-	(e)	-
¹¹ B	0.041	0.085	(e)	-
C	1.19	0.47	(e)	-
N	81.7	85.5	94(g)	1.15
O	8.43	7.2	(e)	-
F	23.46 (f)	5.7	(e)	-
Na	0.648	0.55	(e)	-
Mg	2.86	2.9	-	-
Al	0.710	0.635	0.62	0.87
Si	2.95	0.13/0.99	(e)	-
Cl	12.4 (f)	4.7	(e)	-
K	20.4 (f)	5.8	(e)	-
Ca	-	6.5	(e)	-
Ti	0.145	0.060/0.508	0.24	1.7
V	0.022	0.012/0.04	0.055	2.5
Cr	0.107	0.17/0.2	0.136	1.27
Mn	0.115 (f)	0.13	0.26	2.3
Fe	0.294	0.205	0.34	1.16
Co	0.149	0.14/0.23	0.17	1.14
Ni	4.36	4.52	4.45	1.02
⁶³ Cu	0.354 (d)	0.05	0.49	1.38
Cu	0.286 (f)	0.081	0.34	1.19
Zr	0.002	0.18	(e)	-
Nb	0.074 (f)	1.2	(e)	-
Mo	-	0.13/0.12	(e)	-
Ag	0.072	-	(e)	-
Cd	0.003	-	-	-
Ta	0.0006	-	(e)	-
Au	0.0005	-	0.015	30

(a) Evaluated using Watt fission spectrum, T=1.29, and ENDF/B Version IV (n,α) cross-sections.

(b) Measured value tabulated in Reference 5. When two values are given, the first is from an elemental measurement and the second is derived from isotopic measurements. Large disagreements exist and are not explained.

(c) Preliminary Atomic International helium mass spectrometry measurements on pure elements. Irradiations were made in EBR-II spectra having mean neutron energies of ~ 0.8 MeV.

(d) Evaluated using ENDF/B dosimetry file cross-section (total helium production).

(e) Measurements in EBR-II and Coupled Fast Reactivity Measurements Facility currently underway.

(f) Total helium production cross-section.

(g) The factor used to convert the EBR-II cross-section to the fission spectrum was 3.55 for nitrogen.

- d. The measured values include contributions from impurities such as nitrogen, boron, and phosphorus. Such contributions would be expected to vary radically between materials and perhaps between different lots of the same material.

On the other hand, the ratios in the last column of Table HEDL.13 are not far from unity for most of the elements, indicating that ENDF/B-IV (n, α) cross-sections may be adequate in some cases for helium production estimates in fission reactor spectra. For example, the version IV cross-section for nickel, which is usually the principal helium contributor in irradiated stainless steel, is a considerable improvement over earlier versions which produced ratios as high as ~ 16 .

To make substantial additional improvements, the following steps are necessary:

- a. Self-consistent evaluations or reevaluations of important (n, α) and other helium production cross-sections (including all those in Table HEDL.13 for inclusion in ENDF/B-V. These evaluations should be done using self-consistent nuclear models.
- b. Integral data testing in designated benchmark spectra to make up for the lack of cross-section data for many isotopes. Careful consideration will be required of the effect of nitrogen and other impurities in establishing benchmark measured and calculated spectral averaged elemental cross-sections. Thermal, resonance, fission, fusion, and a few accelerator produced (tailored 1-8 MeV in mean energy)⁽⁶⁾ benchmark spectra have been identified for this data testing.
- c. Evaluations of isotopic cross-sections for some elements, including transmutation products, to allow calculation of effects of very long exposures.
- d. Completion of the measurements at AI of the helium produced in numerous pure elements, compounds, and separated isotopes that were (1) irradiated during three EBR-II dosimetry tests at locations adjacent to HEDL flux-spectral sets, and (2) in other designated LMFBR and CTR benchmark spectra.
- e. Inclusion of the (n, α) cross-sections, together with estimates for other helium production reactions, in a single helium production cross-section file for materials damage and dosimetry uses for the LMFBR and CTR programs. The Cross-Section Evaluation Working Group (CSEWG) has accepted a HEDL recommendation for the development of such a file for inclusion in ENDF/B-V. The CSEWG normalization and standards subcommittee, under the chairmanship of B. L. Leonard of PNL, has been assigned the responsibility of establishing this file because of its previous work in developing the ENDF/B-IV evaluated dosimetry cross-section file.⁽²⁻⁴⁾

HEDL is currently coordinating efforts for the development of the ENDF/B-V helium production file for the LMFBR program. This program work could be extended, with separate DCTR support, to include the needs of the CTR program. Figure HEDL.58 is a flow chart which shows the different elements involved in the development of this helium production file; the involvement of several laboratories is anticipated. The initial phase of this effort will be the identification of (1) all (n, α) reactions of interest for fission and fusion reactor materials, and (2) a number of benchmark spectra for integral data testing of the file.

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FIGURE HEDL.58. Flow-Chart for Development of ENDF/B-V Helium Production File.

3. Expected Achievements

A listing of (n,α) reactions of interest for fission and fusion reactor materials for initial inclusion in the ENDF/B helium production file will be prepared and submitted to the Normalization and Standards Subcommittee of CSEWG.

A joint HEDL-AI paper on the status and need for the development of an ENDF/B helium production file for the LMFBR and CTR programs will be prepared for presentation at the March 1975, conference on Nuclear Cross-Sections and Technology.

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C. LMFBR STRUCTURAL MATERIALS EVALUATION

J. M. Steichen and R. L. Knecht

1. Objective

The objective of this program is to determine the mechanical properties of LMFBR component structural materials for their anticipated service conditions. Results are for use in design confirmation and operations support of FFTF and for materials selection and application in CRBR and other LMFBRs.

2. Accomplishments and Status

An initial characterization of the effect of neutron irradiation on the tensile properties of three FTR structural materials (Inconel 718, Inconel 600, and A-286) has been completed. Materials and irradiation conditions were representative of FTR components and operating conditions. Tensile test conditions included temperatures from room temperature to 1100°F and a strain rate of 3×10^{-5} in./in./sec. Preliminary results from these tests are presented herein.

Specimen irradiations were performed in Row II of EBR-II in subassembly X-195. Irradiation conditions for the test materials were:

Inconel 718 - 760°F to 1.0×10^{22} n/cm² (E>0.1 MeV)
 - 1100°F to 0.9×10^{22} n/cm² (E>0.1 MeV)

Inconel 600 - 760°F to 0.25×10^{22} n/cm² (E>0.1 MeV)

A-286 - 800°F to 0.3×10^{22} n/cm² (E>0.1 MeV)

All specimens irradiated at temperatures of 760°F were irradiated in EBR-II coolant (weeper capsules). The higher irradiation temperatures (800 and 1100°F) were obtained by subcapsule gas gapping and were verified by melt sentinels.

3. Results

a. Inconel 718. The effect of test temperature on the ultimate and 0.2% yield strength of unirradiated and irradiated Inconel 718 is illustrated in Figure HEDL.59. As shown in the figure, strength properties of unirradiated material decrease with increasing test temperature. Neutron irradiation at 760°F to $\sim 1 \times 10^{22}$ n/cm² (E>0.1 MeV) has little effect on either the ultimate or 0.2% yield strength, however, irradiation at 1100°F to the same fluence significantly reduces the 0.2% yield strength and has little effect on the ultimate strength.

The ductility (total elongation and reduction of area) of unirradiated Inconel 718 is influenced by test temperature as illustrated in Figure HEDL.60. Both ductility properties initially decrease with increasing temperature to a temperature of $\sim 450^\circ\text{F}$. From ~ 450 to 600°F substantial increases in ductility are observed and at temperatures above 800°F ductility decreases with increasing test temperature. The enhanced ductility observed in the 600-1100°F temperature range is believed to be the result of dynamic strain aging since pronounced serrations were observed in these stress-strain curves.

Neutron irradiation at 760°F to $\sim 1 \times 10^{22}$ n/cm² (E>0.1 MeV) significantly decreased (by approximately 50%) the total elongation at all test temperatures. Reduction of area

HEDL-75

FIGURE HEDL.59. Strength of Unirradiated and Irradiated Inconel 718.

FIGURE HEDL.60. Ductility of Unirradiated and Irradiated Inconel 718.

values was unchanged by irradiation at 760°F except at the highest test temperature (1000°F) where irradiated values were substantially lower than unirradiated values.

Irradiation at 1100°F appears to slightly enhance the ductility of the subject material at room temperature and 450°F (Figure HEDL.60). At the higher test temperatures (1100 and 1200°F) both total elongation and reduction of area are greatly reduced and approach ~2% at the highest test temperature. Helium embrittlement may be responsible for the lack of high temperature ductility.

b. Inconel 600. Elevated temperature tensile properties of unirradiated and irradiated Inconel 600 are presented in Figures HEDL.61 and .62. Irradiation effects data are presented for an irradiation temperature of 700°F and fluences of 1.3 and 2.5×10^{21} n/cm² (E>0.1 MeV).

The ultimate and 0.2% yield strengths of unirradiated Inconel 600 decrease with increasing test temperature (Figure HEDL.61). Similar behavior is noted for irradiated material. Irradiation hardening of the Inconel 600 slightly increases ultimate strength and significantly increases the yield strength (values for the higher fluence are approximately double unirradiated values) at all test temperatures.

The effect of test temperature on the ductility (total elongation and reduction of area) of Inconel 600 is illustrated in Figure HEDL.62. For temperatures to 800°F ductility remains constant for unirradiated material. Similar behavior is observed for irradiated material, however, total elongation values are slightly reduced as a result of neutron irradiation. For temperatures from 800 to 1200°F ductility of both unirradiated and irradiated material decreases with temperature. The large decreases observed for irradiated material are believed to result from helium embrittlement. Above 1200°F ductility of unirradiated material rapidly increases with temperature. Although tests were not performed on irradiated material in this temperature region, it is anticipated helium embrittlement would severely restrict ductility.

c. A-286. A-286 elevated temperature tensile properties are illustrated in Figure HEDL.63. The heat treatment of the material prior to testing and irradiation included annealing at 1800°F for 30 minutes and precipitation hardening at 1325°F for 16 hours. As shown in Figure HEDL.63, the ultimate and 0.2% yield strength of unirradiated A-286 decreases with increasing temperature. Similar behavior is observed for material irradiated at 800°F to 2.5×10^{21} n/cm² (E>0.1 MeV) and the strength of irradiated and unirradiated material is nearly identical at all test temperatures.

The total elongation and reduction of area of unirradiated A-286 remain essentially constant with temperature to 1000°F and slightly increase with temperature from 1000 to 1200°F. Neutron irradiation slightly enhances total elongation and reduces reduction of area values for test temperatures to 1000°F. At a test temperature of 1200°F both total elongation and reduction of area are reduced by irradiation.

4. Expected Achievements

Testing of Inconel 718 tensile specimens irradiated at 1100 and 1200°F to $\sim 3 \times 10^{22}$ n/cm² (E>0.1 MeV) will be completed during the next reporting period.

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FIGURE HEDL.61. Strength of Unirradiated and Irradiated Inconel 600.

FIGURE HEDL.62. Ductility of Unirradiated and Irradiated Inconel 600.

FIGURE HEDL.63. A-286 Tensile Properties.

D. SUBCRITICAL CRACK GROWTH

L. A. James and R. L. Knecht

1. Objective

The objectives of this study are to determine the subcritical crack growth properties of FFTF/CRBRP structural materials for operating environments, and to perform fatigue failure analyses for structural components.

2. Accomplishments and Status

Linear-elastic fracture mechanics techniques were used to characterize the effect of various elevated temperature operating environments (e.g., air, nitrogen, liquid sodium) and vacuum on the fatigue-crack propagation behavior of reactor structural materials. The materials studies to date include annealed Type 304 stainless steel, annealed Inconel 600, and precipitation-heat treated Inconel 718. Each of the materials investigated were from the same material heats utilized in previous investigations at HEDL: Type 304 stainless (Allegheny Ludlum heat 55697),^(1,2) Inconel 600 (Huntington heat NX-9929-1A,⁽³⁾ and Inconel 718 (Huntington heat 52C9EK).⁽⁴⁾

The tests utilizing an air environment were done in an air circulating furnace. The sodium environment chamber and the vacuum chamber have been described in Reference 1. The same chamber used for vacuum testing was also employed for the nitrogen environment tests. All tests in liquid sodium were conducted in "low oxygen" sodium (1-3 ppm oxygen) except Specimen No. 59 which was tested in "high oxygen" sodium (20-40 ppm oxygen). The vacuum tests were conducted in $6.7\text{--}8.6 \times 10^{-6}$ torr vacuum. The nitrogen environment tests were conducted at one atmosphere in ultra high purity nitrogen ($O_2 < 8\text{ppm}$).

All tests were conducted using feedback controlled electrohydraulic equipment utilizing load as the control parameter. The loading waveforms employed were either sinusoidal or "sawtooth." The tests in nitrogen, vacuum and sodium environments used a fracture face marking technique, employing periodic changes in stress ratio, to obtain the basic crack length versus cycles data. (See Reference 1 for more details). Test results were plotted in the form crack growth rate as a function of the effective stress intensity factor (see Reference 2 for a discussion of K_{eff}).

The results for annealed Type 304 stainless steel tested at 800°F and 1000°F are given in Figures HEDL.64 and 65, respectively. The results for annealed Inconel 600 and precipitation-heat-treated Inconel 718, both tested at 800°F, are given in Figures HEDL.66 and 67, respectively.

It will be noted (Figures HEDL.64 and .65) that, for annealed Type 304, the effects of vacuum and liquid sodium environments are approximately equivalent, and both environments produce fatigue-crack growth rates far lower than does an air environment at the same value of K_{eff} . The results for nitrogen fall between the results for air and for sodium/vacuum, indicating that nitrogen is not as inert as a vacuum but is less aggressive than air. These results are in qualitative agreement with results⁽⁵⁾ for Inconel 718 tested at room temperature in air, nitrogen and vacuum. Crack growth rates that were lower in a nitrogen environment at 1200°F than an air environment at the same temperature have also been noted for annealed

FIGURE HEDL.64. Fatigue-Crack Growth Behavior of Annealed Type 304 Stainless Steel in Air, Vacuum, Nitrogen and Sodium Environments at 800°F. (Air environment data from Reference 7.)

FIGURE HEDL.65. Fatigue-Crack Growth Behavior of Annealed Type 304 Stainless Steel in Air, Vacuum, Nitrogen and Sodium Environments at 1000°F. (Air environment data from Reference 2.)

FIGURE HEDL.66. Fatigue-Crack Growth Behavior of Annealed Inconel 600 in Air and Sodium Environments at 800°F.

FIGURE HEDL.67. Fatigue-Crack Growth Behavior of Precipitation-Heat-Treated Inconel 718 in Air and Sodium Environments at 800°F.

Types 316 and 321 stainless steels.⁽⁶⁾ The results for a nitrogen environment are important since a nitrogen cover gas is used for the primary piping in the FFTF.

The effect of a liquid sodium environment on the crack growth behavior of annealed Inconel 600 and precipitation-heat-treated Inconel 718 is given in Figures HEDL.66 and .67, respectively. As with the previous observations for annealed Type 304 stainless steel, a sodium environment has the effect of significantly reducing the crack growth rates in these two nickel-base alloys relative to an air environment. Resistance to cracking in applications involving sodium thermal transients will therefore be greater than expected on the basis of data obtained by testing in air. The curves for both nickel-base alloys are considered preliminary as additional in-sodium tests are planned for each material.

A small amount of in-sodium testing on Inconel 718 was done at a cyclic frequency of 40 cpm instead of 400 cpm. As may be seen in Figure HEDL.67, there appears to be little or no effect upon the crack growth behavior by reducing the cyclic frequency one decade. Previous results⁽⁴⁾ on this alloy tested in an air environment at 800°F indicated a slight increase in the crack growth rate as the frequency was reduced from 400 cpm to 40 cpm.

3. Expected Achievements

The results described are part of a larger effort to determine the effects of temperature, cyclic frequency, stress ratio, heat treatment, neutron irradiation and operating environments upon the crack growth behavior of reactor structural alloys. Work on these parameters is continuing.

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III. LMFBR STRUCTURAL MATERIALS

T. T. Claudson

A. FFTF/LMFBR WELD MATERIALS PROPERTIES

A. L. Ward, L. D. Blackburn and R. L. Knecht

1. Objective

The objective of this program is to describe the effects of elevated temperatures and fast reactor environments on the mechanical properties of structural base and weld materials for FFTF/LMFBR applications.

2. Accomplishments and Status

Current design rules ⁽¹⁾ for nuclear systems operating at temperatures above 800°F provide for use of the wrought base metal mechanical properties in welded structures but restrict the allowable deformation in the weld region to one-half that permitted for the base metal. Since these rules are based on wrought base materials data, they do not directly account for property variations in and near weldments. The deformation restriction in welds arises from the general knowledge that weld-deposited austenitic stainless steels are often less ductile than the wrought products. Sufficient experimental weld metal property data need to be generated before differences can be treated explicitly and quantitatively.

In view of these general requirements for weld property data, this report presents property trend curves based upon the results of conventional tensile tests conducted over the range from room temperature to 1200°F. The data treatment technique parallels that used by Smith ⁽²⁾ for several austenitic stainless steels and involves establishing ratios of the tensile properties at a given temperature to those at room temperature and then fitting the ratio values to a polynomial expression in temperature.

The ratio trend curves established by Smith for the yield and tensile strength of wrought Type 304 are used directly for comparison with the curves developed here for weld-deposited materials. In addition, since Smith addressed only strength considerations, the present work included formulation of trend curves for total elongation and reduction of area for wrought 304. Data for these curves were taken largely from the same source (ASTM DS5S1) ⁽³⁾ used by Smith; ⁽²⁾ however, no attempt was made to assure one-to-one correspondence with the data used by Smith for the yield and tensile strength treatments. A few test results from a well characterized heat (Al Ht. No. 55697) of 304 (3/8 inch diameter rod) were also included.

Detailed descriptions of the weldments, materials and specimens are given elsewhere ⁽⁴⁾ along with the experimental data used in this analysis. For evaluation purposes, the data were arbitrarily divided into three groups according to filler metal composition: 308/308L Group; CRE 308 Group (CRE denotes controlled residual elements -- B, P and Ti); and 16-8-2 (Cr-Ni-Mo) Group. A fourth group designated All-Weld includes all of the data from the three groups above plus data from a 316 weld.

The procedures for establishing the behavioral trend curves began simply by computing the ratio values for all of the elevated temperature test results. These ratio values

were then fitted by a computer program to a polynomial of the form:

$$R_p = A_0 + A_1T + A_2T^2 + \cdots A_iT^n$$

by the least squares regression method. The program provided values of the coefficients and the standard error of the estimate of R_p . Since the ratio value at room temperature is defined as unity, a number of such values were input in order to force the curve to unity at 75°F. Adequacy of the fit was judged on the basis of a combination of minimizing the value of the standard error of the estimate of R_p (S_y) and subjective visual examination of the resulting curves.

3. Results

Room Temperature Properties. A summary of the room temperature properties is given in Table HEDL.14. The maximum and minimum values are based upon observed values and not upon any statistical evaluation. Because the 308/308L group contains property values for both as-welded and heat treated materials it is divided into two parts: as-welded only and all-308/308L-group data. It can thus be seen that the various heat treatments reduced the mean value by 10% and yielded a minimum value nearly 10 ksi lower than that of the as-welded-only portion.

TABLE HEDL.14
ROOM TEMPERATURE PROPERTY SUMMARY

Data Group	Value	σ_y	σ_u	ϵ_u	ϵ_T	RA	No. of Tests
308/308L (As-Welded Only)	Mean	55.7	84.2	31.6	39.3	64.0	12
	Std. Dev.*	8.3	5.5	6.5	7.0	8.4	
	Min.	45.2	76.1	22.3	31.1	46	
	Max.	70.7	92.7	41.0	54.0	77	
All 308/ 308L	Mean	50.1	83.8	35.7	41.7	59.7	24
	Std. Dev.*	9.7	5.5	7.9	7.5	9.1	
	Min.	35.5	75.5	22.3	31.1	40	
	Max.	70.7	92.7	49.3	54.2	77	
CRE 308	Mean	66.8	92.2	33.1	37.3	60.5	4
	Std. Dev.*	7.5	3.8	1.7	3.6	6.0	
	Min.	58.1	88.4	30.6	32.6	53	
	Max.	76.2	97.1	34.2	40.9	67	
16-8-2	Mean	49.2	90.0	44.0	48.3	50.3	3
	Std. Dev.*	8.6	4.1	7.9	9.0	15.7	
	Min.	45.9	85.4	38.2	42.9	38	
	Max.	61.6	93.3	53.0	58.7	68	
All Welds	Mean	52.8	85.5	36.1	41.7	58.9	31
	Std. Dev.*	10.8	6.0	7.7	7.5	9.6	
	Min.	35.5	75.5	22.3	31.1	38	
	Max.	76.2	97.1	53.0	58.7	77	

*Std. Dev. = $[\sum_i (x_i - \bar{x})^2 / (n-1)]^{1/2}$ where x_i = property values, $\bar{x}$ = arithmetic mean value, and n = sample size or number of tests.

Wrought Type 304 Evaluation. The yield and tensile strength trend curves for the Type 304 are those of Smith⁽²⁾ and those for total elongation (ϵ_T) and reduction of area (RA) were developed in the present work. The analysis of the wrought Type 304 ductility is summarized in Table HEDL.15 along with the results of Smith's analysis of the strength. Two ϵ_T ratio values are given at each temperature because of an anomalous, but distinct separation of the data into two populations.

The reduction of area evaluation (Table HEDL.15) extends only to a temperature of 1100°F because there were considerably fewer data available at higher temperatures and because the existing data indicate a very sharp reduction between 1100 and 1200°F. This had the effect of introducing a substantial difference between the regression curve and the data in the region near the apparent discontinuity.

Weld Metal Evaluation. The results of the weld metal property analysis are also summarized in Table HEDL.15. The values listed were taken from the regression curves at 100°F intervals.

The yield strength ratio curves (Figure HEDL.68) for the weld materials data groups are quite similar in form and magnitude despite the variations in materials, welding procedures, material conditions, and data sample size. For this ratio, the all-weld trend curve could be used without introducing differences of more than 10% in the ratio values even at 1200°F. In all cases, the weld-deposited austenitic stainless steels appear to maintain a higher fraction of their room temperature strengths at elevated temperatures than does the wrought Type 304 in these short-term tests.

The ultimate tensile strength ratio trend curves for the weld metal data groups are basically similar in both shape and magnitude (Figure HEDL.69) up to 1000°F, but appear to diverge at higher temperatures. Below 1000°F, the all-weld curve could be used without introducing differences of more than 5%. At higher temperatures, however, the curves for the 308/308L and CRE 308 materials decrease more rapidly than does the curve for 16-8-2 materials and adoption of the all-weld trend curve leads to differences in ratio values as large as 33%. This maximum difference occurs at 1200°F where there were no data for the 308/308L group and the ratio value represents a 100°F extrapolation of the polynomial expression.

The uniform elongation values used in this study are those engineering strain values which correspond to the point of maximum load on the stress-strain curves (Figure HEDL.70). The ratio trend curves shown in Figure HEDL.72 do not include curves for the 304 because uniform elongation data were not included in the data source used (ASTM D551). In all the weld data groups, the trend curves exhibit a minimum and a maximum. This behavior could be the result of the mathematical curve-fitting method rather than a real physical phenomenon, particularly in view of the limited data at low temperatures. Uniform elongation trend curves are essentially identical for the 308/308L and CRE 308 groups, but the curve for 16-8-2 material is significantly lower. In this case, the all-weld trend curve introduces rather large differences (i.e., up to 50%) in ratio values for the 308-type groups at 1200°F and significant differences (i.e., up to 23%) in ratio values for the 16-8-2 material throughout much of the temperature range.

TABLE HEDL.15

LEAST SQUARES POLYNOMIAL REGRESSION ANALYSIS RESULTS
(PROPERTY RATIOS VS TEST TEMPERATURE)

Property	S _y *	Temperature, °F											
		100	200	300	400	500	600	700	800	900	1000	1100	1200
<u>308/308L</u>													
	.0846	.982	.919	.870	.831	.800	.773	.747	.718	.682	.637	.580	.506
σ _y	.0540	.973	.886	.832	.802	.788	.779	.767	.744	.699	.624	.511	.349
σ _u	.1355	.968	.851	.755	.679	.624	.590	.575	.582	.608	.656	.723	.812
ε _T	.1220	.942	.762	.652	.598	.582	.590	.606	.614	.600	.547	.440	.263
ε _u	.1287	.988	.940	.900	.869	.848	.835	.831	.836	.850	.874	.906	.947
RA													
<u>CRE 308</u>													
	.0492	.981	.918	.865	.821	.784	.751	.722	.695	.668	.638	.606	.568
σ _y	.0174	.970	.876	.811	.770	.745	.730	.718	.702	.676	.632	.564	.464
σ _u	.0876	.952	.802	.700	.638	.606	.597	.602	.611	.617	.610	.581	.522
ε _T	.1060	.938	.743	.627	.572	.560	.575	.598	.612	.601	.545	.428	.233
ε _u	.0922	.994	.982	.963	.940	.918	.901	.892	.896	.917	.958	1.020	1.120
RA													
<u>16-8-2</u>													
	.0843	.992	.949	.906	.864	.822	.782	.742	.703	.664	.626	.589	.553
σ _y	.0372	.971	.877	.813	.773	.751	.740	.734	.727	.711	.682	.632	.556
σ _u	.0816	.954	.800	.685	.604	.552	.523	.512	.516	.527	.542	.556	.562
ε _T	.0774	.944	.758	.627	.543	.495	.475	.473	.482	.490	.491	.473	.429
ε _u	.1414	1.022	1.077	1.117	1.144	1.156	1.154	1.138	1.109	1.065	1.006	.934	.848
RA													
<u>ALL-WELD</u>													
	.0780	.983	.926	.878	.838	.804	.773	.744	.714	.683	.648	.606	.560
σ _y	.0542	.973	.894	.839	.804	.782	.766	.751	.730	.697	.647	.572	.466
σ _u	.1310	.954	.804	.697	.626	.586	.571	.574	.588	.609	.630	.644	.646
ε _T	.1342	.943	.765	.652	.590	.567	.568	.581	.593	.589	.557	.484	.355
ε _u	.1518	.996	.963	.936	.915	.900	.892	.889	.893	.903	.919	.942	.970
RA													
<u>WROUGHT 304</u>													
	-----	.96	.83	.75	.69	.65	.61	.59	.56	.54	.52	.49	.47
σ _y	-----	.97	.86	.80	.78	.77	.77	.77	.76	.74	.70	.63	.55
σ _u	.036/.048	.975/.960	.880/.820	.790/.700	.735/.635	.710/.610	.710/.610	.730/.610	.745/.610	.740/.605	.700/.595	.635/.565	.550/.540
ε _T	.043	.994	.968	.945	.927	.910	.895	.882	.875	.867	.865	.860	.857
RA													

$$*S_y = [\sum (d^2) / n]^{1/2}$$

where d = deviation of actual values from curve, and
n = sample (number of data).

FIGURE HEDL.68. Yield Strength Ratio Trend Curves for Weld-Deposited Stainless Steels and Wrought Type 304.

FIGURE HEDL.69. Tensile Strength Ratio Trend Curves for Weld-Deposited Stainless Steels and Wrought Type 304.

FIGURE HEDL.70. Uniform Elongation Ratio Trend Curves for Weld-Deposited Stainless Steels.

FIGURE HEDL.71. Total Elongation Ratio Trend Curves for Weld-Deposited Stainless Steels and Wrought Type 304.

FIGURE HEDL.72. Reduction of Area Ratio Trend Curves for Weld-Deposited Stainless Steels and Wrought Type 304.

Trend curves for total elongation are quite similar for the 308/308L and CRE 308 materials up to 900°F (Figure HEDL.71). At higher temperatures, the curves for these groups diverge markedly. Again, the trend curve for 16-8-2 material is lower than the other two over the range from 600 to 1000°F. As a result, the all-weld trend curve leads to potentially significant differences in ratio values, but these differences are generally smaller than for the case of uniform elongation. In general, the weld materials trend curves compare favorably with the lower wrought 304 curve. Over the entire temperature range, there is remarkable similarity between the curves for the CRE 308 and the lower wrought 304 group, with the largest difference of 3.5% occurring at 1200°F. At and below 1000°F, there is also little difference (6.8% maximum) between the all-weld and the lower wrought 304 curves although the difference reaches nearly 20% at 1200°F largely because of the effect of including the 308/308L data.

Reduction of area trend curves for the 308/308L and CRE 308 groups are only slightly different, and are in reasonable agreement with the 304 curve up to 800°F (Figure HEDL.72). At higher temperatures, however, the two weld metal curves increase significantly, while the 304 curve does not. The 16-8-2 group trend curve lies significantly higher than those of the other welds, but the scatter in the ratio values was quite large for this material and the shape of the resulting trend curve may not be realistic. Differences between the all-weld trend curve and the individual group curves are not large (i.e., generally less than 7%) for the 308/308L and CRE 308 materials, but are more significant (i.e., up to 23%) for the 16-8-2 group.

Discussion. The trend curve analysis shows differences among the three weld groups in the ultimate tensile strength and the ductility parameters. The 308/308L and CRE 308 materials exhibit basically similar trend curves for all parameters, which is not unexpected since the compositional differences between these groups are primarily in the minor elements. It is quite possible that the differences which are indicated for these groups stem in part from the inclusion of a greater range of welding parameters and material conditions in the data sample for the 308/308L material. Differences between the 308-type groups and the 16-8-2 group are somewhat more pronounced, but once again variations in welding parameters may contribute to the apparent differences, particularly in view of the smaller sample size for the 16-8-2 group. The good agreement among the yield strength ratio trend curves suggests that perhaps the effect of temperature on the early stages of plastic deformation is quite similar for weld-deposited austenitic stainless steels in general, with recognizable differences between various welds arising at larger deformations associated with instability and fracture.

The most prominent difference between ductility parameter trend curves for weld materials and wrought 304 is the increase in ratio values for total elongation (308/308L group) and reduction of area (308/308L group and CRE 308 group) at temperatures above 800°F. Type 304 stainless steel frequently exhibits a decrease in fracture ductility (particularly reduction of area) at temperatures above about 1100°F accompanying the onset of intergranular fracture. However, the possibility that the increases in weld ductility ratios over the range from 800 to 1200°F are associated with a suppression of intergranular failure has not been clearly demonstrated.

The interpretation and utilization of the property ratio trend curves should be made with a recognition of the variability or scatter in the experimental data. One measure of data variability is the standard error of the estimate of the property ratio trend curve, S_y . The statistical quantity, S_y , relates to the regression curve in a manner which is exactly analogous to the relationship of the standard deviation to the arithmetic mean of a data sample. As such, S_y is a measure of the dispersion, variability or scatter about the line of regression.

Values of S_y given in Table HEDL.15 clearly show that less variation is observed in the strength parameters than in the ductility parameters. Also, the S_y values for ultimate strength ratio curves are consistently lower than values for yield strength curves, indicating greater variation in the latter trend curves. Comparing total elongation and reduction of area trend curves for weld materials with those for wrought 304 (Table HEDL.15), substantially higher S_y values are obtained for welds, reflecting a greater variability in the ductility parameter for these materials. Although results for S_y were not reported by Smith⁽²⁾ for yield and ultimate tensile strength trend curves for 304, we might also expect lower S_y values and less variability for the wrought product than for welds.

Data variability can be viewed in a somewhat different light by examining the accuracy with which the trend curve approach correlates the absolute property values. To do this, we selected the trend curves for the 308/308L group and the CRE 308 group, and for each data set computed elevated temperature property values using the ratio values and the measured room temperature properties. These predicted property values were then compared with

experimentally observed values. For the CRE 308 group, the correlation is good for both yield strength and ultimate tensile strength, predicted values falling in general within ± 4000 psi of measured values. Comparison of predicted and measured total elongation values showed rather large variations; predicted values generally falling within $\pm 7.5\%$ (on an absolute basis) of measured elongation values. For the 308/308L group, predicted yield strength values generally fell within $\pm 8,000$ psi of measured values, while predicted ultimate tensile strengths were within about $\pm 6,000$ psi of experimental results. In the case of total elongation, predicted values are within about $\pm 10\%$ (on an absolute basis) of measured values.

It is significant that the CRE 308 group produced the lowest overall S_y values and showed the closest agreement between measured and predicted properties. This is the only group in the study which represents a single material (CRE 308) and a single weld process (SMA) with fairly similar welding parameters. The degree of departure from a perfect correlation was substantially greater for all 308/308L properties than for the CRE 308. While this result is not surprising in view of the greater variation in weld process and parameters, it serves to emphasize the difficulties associated with establishing broadly applicable weld metal property specifications and thus points to a need for additional work in this area. Nevertheless, the present work indicates that the property ratio trend curve approach provides a useful degree of correlation of the elevated temperature properties of an important group of materials.

4. Expected Achievements

The tensile property analysis presented here completes this phase of study conducted under the LMFBR Structural Materials Program, although as additional new data are generated, the curves will be updated. Current program goals include a behavioral description of the creep properties of weld deposited austenitic stainless steels, however, near term emphasis will be placed on analysis of postirradiation data taken from materials irradiated in the FTR Component Materials Irradiation Experiment and on extending irradiation effects studies to reference and candidate materials for the CRBR demonstration plant.

5. References

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B. SPATIAL DEPENDENCE OF VOID GROWTH RATES IN THE IRRADIATION OF THIN FOILS --
THEORY AND CALCULATIONS

G. L. Guthrie, J. L. Straalsund and T. K. Bierlein

1. Objective

The objective of this study is to present an analysis approach which indicates a deeper penetration depth of the surface boundary effect.

2. Accomplishments and Status

The existence of a void depleted region near grain boundaries or external surfaces of irradiated specimens has been noted by several observers.⁽¹⁻³⁾ The increased use of high voltage electron microscope (HVEM) irradiations to provide short time simulations of reactor exposures has stimulated additional interest in this phenomenon. Imaging considerations in the electron microscope dictate the use of thin foils for the bombardments, and under these conditions, the depleted region occupies a non-negligible fraction of the total specimen volume.

Papers previously published by other authors have considered the theoretical calculations of both steady state^(4,5) and time dependent⁽⁶⁾ vacancy spatial distributions near boundary surfaces in irradiated foils. This has been done under conditions involving both sink-free environments (4-6) and environments containing unbiased sinks.^(5,6)

The present study deals with the steady state spatial distribution of the "growth fluid" ($D_V C_V - D_I C_I$) near the boundary of a thin foil during irradiation, considering both biased and unbiased sinks, together with provision for recombination. The important result is that the analysis indicates a deeper penetration depth of the surface boundary effect, compared to previous steady-state work that concerned surface influence on vacancy concentrations.

In the expression for the "growth fluid" above, the D's are diffusivities, the C's are point defect concentrations, and the subscripts refer to vacancies and interstitials. The significance of $D_V C_V - D_I C_I$ (hereinafter referred to as ξ) is that $-\nabla \xi$ gives the direction and net flux of vacancies across a surface. That is, it gives vacancy flow minus interstitial flow. In a sea of uniformly spaced sinks having surface equilibrium values of $C_V = 0$ and $C_I = 0$, the net rate of flow of vacancies into sinks is given by a geometrical factor times the local average value for the irradiated continuum. The value of ξ is important in considering both growth rates and nucleation probabilities for voids.

If we neglect thermal equilibrium concentrations of point defects, the governing equations for the steady state point defect concentrations may be written in simplified form as:

$$\nabla^2 V - SV - RIV + G = 0 \quad (1)$$

$$\nabla^2 I - S'I - RIV + G = 0 \quad (2)$$

where $V = D_V C_V$ and $I = D_I C_I$. S is a coefficient for vacancy capture by voids and dislocations. Its magnitude depends on void size and density, and also on dislocation density. The vacancy capture rate per unit volume due to capture by dislocations and voids is given by SV . S' is a similar coefficient for interstitials. G is the generation rate per unit

volume, which, for this note, we assume to be independent of position, and RIV is the recombination rate per unit volume.

Since ξ is the quantity of interest, we subtract Equation (2) from Equation (1), obtaining:

$$\nabla^2 \xi = S\xi = -(S'-S)I. \quad (3)$$

Equation (3) is extremely useful both for purposes of insight and for purposes of calculation, where its use reduces roundoff errors which would occur in a direct application of Equations (1) and (2) to obtain V , I , and then $(V-I)$ by direct subtraction.

Reading from left to right in Equation (3), we note that the three terms represent diffusion, trapping and a "source," or driving term. We note that the driving term, $-(S'-S)I$ would be zero in Equation (3) if one incorrectly ignored dislocation bias, and this would lead to the erroneous result that $\xi = 0$ everywhere.

For the sake of comparison between the C_v and ξ equations, we note that in Equation (1) the damping coefficient is $(S+RI)$ while in Equation (3) the damping does not include recombination, but is simply S and is therefore smaller. Consequently, in the vicinity of a perturbing surface the vacancy concentration will dampen out to the bulk value in a much shorter distance than the quantity ξ . Also in the case of ξ , since the damping distance $(S)^{-1/2}$ is dependent only on the sink density, it is therefore independent of displacement rate and temperature except insofar as they affect the sink density in an actual irradiation.

The comparison of the depth of influence on the surface on C_v and on ξ is shown in Figures HEDL.73 and also in HEDL.74. In each of these figures, both C_v and ξ are plotted vs distance from the surface using reduced coordinates for a particular set of parameter values. In each case, the calculated values are from a digital computer result of a one-dimensional finite difference solution of Equations (1) and (2), where the $I(x)$ solution was then used in Equation (3) to obtain the digital solution for $\xi(x)$. In each of the two figures, it is evident that the surface effect penetrates deeper for ξ than for C_v . In Figure HEDL.73, with a dislocation density of 10^8 lines/cm², we find that the surface effect penetrates to the center of the foil, where ξ attains only 0.6 of the value it would have in an infinitely

FIGURE HEDL.73. A Comparison of the Depth of Influence of the Surface Perturbation on Reduced C_v and Reduced ξ .

FIGURE HEDL.74. A Comparison of the Depth of Influence of the Surface Perturbation on Reduced C_v and Reduced ξ for a Slightly Higher Dislocation Density.

thick bulk material. However, the extreme depth of surface influence is due to the low value assumed for the dislocation density, and such a low dislocation density would only be found in an HVEM irradiation during the first few seconds of an irradiation of non-preconditioned material. In Figure HEDL.74, with the higher dislocation density of 10^{10} cm^{-2} , we find that ξ has attained 0.9 of the bulk value at a depth which is 1/6 of the foil thickness. A dislocation density of 10^{11} cm^{-2} would result in ξ attaining 0.9 of the bulk value at a depth which is approximately 1/20 of the foil thickness, with all parameters other than dislocation density keeping the values shown in Figure HEDL.74.

It is possible to find a simple approximate analytical solution for ξ that is reasonably accurate. The method of solution is to ignore the $\nabla^2 C_v$ and $\nabla^2 C_I$ terms and solve Equations (1) and (2) algebraically for the bulk steady-state value of C_v and C_I . The bulk steady-state value of C_v is then used in Equation (2) (including the ∇^2 term) to get a position dependent solution $I(x)$, where x is the distance from the foil surface. The resultant expression for $I(x)$ is introduced into Equation (3), which is solved for the steady state value of $\xi(x)$. It is found that:

$$\xi(x) = \frac{F}{S} + \frac{F}{(A-S)} \cdot \frac{\cosh(x\sqrt{A})}{\cosh(x_{1/2}\sqrt{A})} - F \left(\frac{1}{S} + \frac{1}{A-S} \right) \cdot \frac{\cosh(X\sqrt{S})}{\cosh(x_{1/2}\sqrt{S})} \quad (4)$$

where $S = \rho_D Z_v + \rho_{\text{void}} Z_{\text{NT}}$ and $A = S' + RV(\text{bulk})$ and $F = G\rho_D (Z_I - Z_v)/A$, where Z_I and Z_v are the Bullough dislocation point defect capture factors for interstitials and vacancies, ρ_D is the dislocation density, Z_{NT} is a similar capture factor for point defects for neutron traps (voids) defined by: trapping rate per unit volume = Z_{NT} (point defect density) ρ_{void} . In the relations above, ρ_{void} is the void density and $x_{1/2}$ is the distance from the free surface to the center of the sample. In the solution of the $I(x)$ equation and also in the subsequent solutions of the $\xi(x)$ equation, it is assumed that $I(x) = 0$ and $\xi(x) = 0$ at the free surfaces. When the thermal equilibrium terms are included in Equations (1) and (2), the mathematics are similar to that shown, provided that $I(x)$ is replaced by $I(x) - I_{\text{THERMAL EQUILIBRIUM}}$ in Equation (3) and $\xi(x)$ is replaced by $\xi_{\text{THERMAL EQUILIBRIUM}}$. The analytical method just described is only valid if $(S + RI_{\text{bulk}})^{-1/2} \ll \text{foil thickness}$, as otherwise C_v is not well

approximated by the bulk value in the zero order iteration, due to the large region of surface influence on C_v .

For a wide variety of values of the parameters (ρ_D , t , foil thickness, G , etc.) the agreement between the analytical approximation of Equation (4) and the complete finite difference digital solution for ξ is excellent. For the parameter values of Figure HEDL.73, the maximum discrepancy in ξ by the two methods of calculation is 1%. Agreement is excellent if $RI_{bulk} \gg S$ and $(S + RI_{bulk})^{-1/2} \ll \text{foil thickness}$.

In summary then, it is quite evident that the effect of the surface on void nucleation and growth can only be properly assessed by including biased sinks and considering the spatial dependence of $\xi(x)$ rather than $C_v(x)$. This treatment of the problem results in a deeper surface effect, and a different dependence on generation rate, temperature and microstructure.

4. References

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C. EVALUATION OF 20% COLD-WORKED 316 SWELLING CORRELATION

F. A. Garner, G. L. Guthrie and T. K. Bierlein

1. Objective

The objective of this work is to develop design correlations for radiation induced swelling in the reference cladding and duct material. The correlations are under continuous review and are updated periodically as new data become available.

2. Accomplishments and Status

The current design correlation⁽¹⁾ for swelling of 20% CW stainless steel envisions the development of a constant swelling rate after a nonlinear incubation period, as shown in Figure HEDL.75. The steady-state swelling rate R is temperature dependent as are the parameters τ and α , which represent the incubation fluence and rate of transition to the linear swelling behavior. The following correlations are used to describe these parameters:

$$R(T) = \exp \left(-88.5499 + 0.531072T - 1.24156 \times 10^{-3} T^2 \right. \\ \left. + 1.37215 \times 10^{-6} T^3 - 6.140 \times 10^{-10} T^4 \right)$$

$$\tau(T) = \exp \left(-16.7382 + 0.130532T - 3.81081 \times 10^{-4} T^2 \right) \\ + 5.51979 \times 10^{-7} T^3 - 3.26491 \times 10^{-10} T^4$$

$$\alpha(T) = -1.12 + 6.89 \times 10^{-3} T$$

where T is the centigrade temperature. Tabulations of these parameters are given in

FIGURE HEDL.75. Form of Equation Currently Used to Describe Swelling Dependence of 300 Series Stainless Steels.

Table HEDL.16. Note that the incubation parameter $\tau(T)$ in general represents a lower limit since the data field used at that time did not exhibit swelling at all temperatures. The uncertainty bond for this correlation ranged from the nominal equation to 50% of the nominal.

Since the development of this correlation, the relevant data field has expanded to include the points tabulated in Table HEDL.17. All of these data are from air melted heats, which have less stringent specifications than do the vacuum melted FTR first-core and vendor qualification cladding heats. While the addition of small amounts of impurities has been shown to retard swelling in pure metals such as copper, nickel and aluminum, no difference in swelling between heats melted in air and vacuum has yet been demonstrated for steels. Removal of all impurities has led to greatly enhanced swelling in neutron irradiated solution annealed 316 steel, however.⁽²⁾

Impurities have been postulated to affect swelling in two ways. The incubation fluence may be changed, the steady-state swelling rate may be altered or a combination of both effects may occur. Electron simulation experiments have shown that the steady-state swelling rates which develop are generally independent of the heat identity, but that the incubation period varies strongly with local microstructure.⁽³⁻⁴⁾ A typical series of comparative irradiations at 650°C is shown in Figure HEDL.76 for FTR tube and duct steels and Program

TABLE HEDL.16
20% CW 316: CURRENT DESIGN SWELLING CORRELATION
TEMPERATURE DEPENDENT PARAMETERS

Temperature (°C)	R	τ	α
350	0.0058	2.31	1.29
375	0.0023	3.57	1.47
400	0.0691	4.35	1.64
425	0.165	5.03	1.81
450	0.352	5.72	1.98
475	0.539	6.17	2.16
500	0.771	6.35	2.33
525	0.968	6.21	2.50
550	1.077	5.68	2.67
575	1.068	4.80	2.84
600	0.943	3.69	3.02
625	0.737	2.53	3.19
650	0.502	1.50	3.36
675	0.294	0.76	3.53
700	0.144	0.31	3.71
725	0.0574	0.10	3.88
750	0.0179	0.026	4.05

TABLE HEDL.17
COMPILATION OF RECENT 20% CW 316 SWELLING DATA

Material	Source	Irradiation Temperature, (°C)	Fluence, 10^{22} n/cm^2 ($E > 0.1 \text{ MeV}$)	$\Delta V/V_0$ (%)
20% CW 316 Drawn Tubing Heat #M2783	X-098	454	8.6	0.13
		454	8.4	0.05
		535	8.9	0.42
		535	8.6	0.86
		609	8.8	2.30
		609	8.6	1.60
		689	9.4	0.01
		689	9.2	-0.10
27% CW 316 Swaged Rod Program Heat	B-92 (X-157)	390	5.6	0.31
		430	7.6	0.83
		460	6.6	0.42
		625	9.5	1.42
		685	9.9	0.46
		750	10.1	-0.03
		810	10.1	-0.02
20% CW 316 Rolled Sheet Heat #34357	X-100	500	8.0	-0.1
		600	8.0	3.3
20% CW 316 DO Heat (AI)	X-100	500	6.6	-0.10
		600	6.6	-0.40

FIGURE HEDL.76. Comparative Electron Irradiations of Three Heats of 20% CW 316 at 650°C.

Heat (air melted). As shown previously⁽³⁾ there is a range of τ values for each material, so no assessment may be made from these data as to which material has the superior swelling resistance.

It seems quite possible that the range of incubation parameters in a given material will be dependent on composition and thermomechanical treatment, both of which affect the local microstructure. For neutron irradiations, there will be some bulk-integrated incubation parameter for each material

$$\tau_i(T) = \frac{\int \tau_i(T, dV) dV}{\int dV}$$

where dV is the individual volume element and i designates the material identity.

If we assume a normal distribution of $\tau(T)$ values throughout the material then the probability of any volume having a given (T) is

$$P_i(\tau) = \frac{1}{\sqrt{2\pi}\sigma_i} \exp \left[-\frac{(\tau - \tau_i(T))^2}{2\sigma_i^2} \right]$$

As shown in Figure HEDL.77, two different heats might have macroscopic or bulk averaged values of $\bar{\tau}_i$ which are different, even though there may be overlap in the microscopic τ values.

FIGURE HEDL.77. Differences in Bulk Swelling Behavior Arise Due to Varying Microscopic Distributions of Incubation Period.

3. Results

It is possible to analyze the data in Table HEDL.17 for consistency with the current prediction, providing one is willing to accept the quoted values of fluence and temperature. The determination of temperature usually represents the largest uncertainty for in-reactor experiments (see also Figure HEDL.78). Figure HEDL.77 shows that the data field cannot be easily normalized due to the wide variation of fluences. We can reduce the complexity of the problem by visualizing the data in τ -space rather than swelling space. This has the benefit of automatically normalizing all data, removing the dimension of fluence.

The following guidelines were used in this analysis:

- (1) $R(T)$ and $\alpha(T)$ would be preserved from the current correlation.
- (2) The new data points would be fit by finding τ^* values necessary to reproduce the observed swelling at that temperature and fluence.
- (3) These τ^* values are then analyzed for trends.

Figure HEDL.79 shows the new data plotted in τ -space, as well as the data used by Laidler (in as yet unpublished data) in developing the previous $\tau(T)$ correlation. With the exception of the B-92 data points below 500°C, all data lie above the current correlation, which was expected to be a lower bound. With an additional exception of the AI data point at 600°C, all points lie in a relatively tight band which is best defined by the X-098 data, the only

FIGURE HEDL.78. Graphical Compilation of Cold-Worked 316 Swelling Data Shown in Table HEDL.17.

FIGURE HEDL.79. Swelling Data of Table HEDL.17 Reduced to τ -Space.

data set that spans the full temperature range. Note that this reduction technique has reconciled apparently large differences in the X-098 and D0 swelling data. Ignoring the exceptions, a curve labeled τ_{mod} was placed through the data, and has the following functional form:

$$\tau_{\text{mod}}(T) = 2.57262 \times 10^2 - 2.39381T + 8.16677 \times 10^{-3}T^2 \\ - 1.18508 \times 10^{-5}T^3 + 6.21367 \times 10^{-9}T^4 .$$

The form of this curve requires some comment since a nearly straight line placed around $\tau = 8.0$ for all temperatures would probably fit as well. The recent data of Brager⁽⁵⁾ indicate that there is a peak in the incubation period of 20% CW 316 around 500°C and that above 600°C it again becomes difficult to nucleate voids. Thus the shape of the curve is consistent with experimental observations, whereas a constant τ is not.

The B-92 data below 500°C are completely inconsistent with this curve and even become negative below 400°C. This material has a higher level of cold-work however (27%), and there is some concern about the actual irradiation temperatures. The agreement at higher temperatures may be due to the recovery of 20 and 27% cold-work to the same level. Note that the $[\tau_{\text{mod}}(T) - 2.0]$ curve is in rough agreement with the AI data point and the earlier data of Laidler, and forms a lower bound estimate of (T). Reconciliation of the low temperature B-92 data would require $[\tau_{\text{mod}}(T) - 4.0]$.

The viability of the modified τ equation is shown in Figure HEDL.80, where data and predictions for the X-098 data are compared. A more pertinent question is whether the design prediction for FTR and Demonstration Plant are changed substantially using this approach. Shown in Figure HEDL.81 are the comparative fluence dependencies of swelling at 600°C illustrating the range of incubation periods indicated by this data set. Figures HEDL.82 and .83 show the temperature dependence of the three predictions at FTR and Demonstration Plant goal fluences. The range of predictions is smaller than the currently employed uncertainty band, which ranges from the nominal value to 50% of that value.

Note that Figure HEDL.82 assumes constant fluences across the entire temperature range which is of course inconsistent with the actual situation in-reactor. The consideration of flux and temperature gradients may lead to predictions which have greater variations than those shown in Figure HEDL.82. These variations will be most obvious on the ends of the FTR core, where the accumulated fluences will be closest to the incubation value. This subject will be explored further in the next reporting period.

4. Expected Achievements

Anticipated additional data will be reviewed and incorporated into the analysis. The impact of the alternate incubation parameter equations and actual flux and temperature profiles on component swelling will be explored.

5. References

1. Nuclear Systems Handbook, TID-26666.
2. J. M. Leitnaker, et al., "The Effect of Minor Constituents on Swelling in Stainless Steel," J. Nuclear Materials, 49, 57-59 (1973).
3. F. A. Garner, et al., "Comparative Electron Irradiations of FTR First Core Tubing," p. D-29, HEDL-TME 74-2.

FIGURE HEDL.80. Comparison of X098 Swelling Data with Predicted Values. (The dark dot represents the actual data point and the open dot is the value predicted by the new correlation for the quoted fluence.)

FIGURE HEDL-81. Comparison of Swelling Predictions at 600°C.

FIGURE HEDL.82. Comparison of Swelling Predictions at Constant Fluence.

FIGURE HEDL.83. Comparison of Swelling Predictions at Constant Fluence.

4. F. A. Garner, et al., elsewhere in the HEDL part of this report.
5. H. R. Brager and J. J. Laidler, "Swelling of CW Type 316 Irradiated to High Neutron Fluences," p. D-40, HEDL-TME 74-2.

D. DEVELOPMENT OF SOLUTION ANNEALED 316 SWELLING CORRELATION

F. A. Garner and T. K. Bierlein

1. Objective

The objective of this work is to develop, review and revise design correlations for radiation induced swelling in fast reactor structural materials. As additional neutron or charged particle simulation data become available, the data are reviewed for impact on previously developed correlations.

2. Accomplishments and Status

Scope. A new swelling equation has been developed in response to a request from the working group on the LIFE-III fuel pin calibration effort. This equation utilizes the bilinear swelling law that has been observed in electron simulations.⁽¹⁾ The previous correlation was of nonlinear form and was derived from a data base published in late 1971.⁽²⁾

Additional swelling data compiled by mid-1973 by J. J. Laidler and J. L. Straalsund was used to expand and reanalyze the swelling behavior of solution annealed 316 stainless steel. Very recently, additional data have become available and used to test the predictive capability of the correlation supplied for the LIFE-III calibration effort.

Table HEDL.18 lists the data set employed⁽²⁾ in the development of the previous correlation and Table HEDL.19 contains the later compilation. Unfortunately the combined contents of these two tables are insufficient to allow division of the data into fine temperature intervals. This places a limitation on our ability to provide a full description of the temperature dependence. A compromise between statistics and temperature intervals resulted in the choice of four increments of 50°C each between 400 and 600°C. The resultant data sets are shown in Figures HEDL.84 through .87. The 20% cold-worked 316 data available at that time are also shown for comparison. While the annealed data sets are not very extensive they are obviously more developed than the cold-worked data. On each of Figures HEDL.84 through .87 are shown estimates of the steady-state swelling behavior.

The form of the equation chosen for use as the design equation is that used previously for 20% CW 316⁽³⁾ and annealed 304,⁽⁴⁾ as follows:

$$\frac{\Delta V}{V} = R \left\{ \phi t + \frac{1}{\alpha} \ln \left[\frac{1 + \exp [\alpha(\tau - \phi t)]}{1 + \exp (\alpha \tau)} \right] \right\},$$

where: $\Delta V/V_0$ = swelling in percent

ϕt = neutron fluence ($E > 0.1 \text{ MeV}$), in units of 10^{22} n/cm^2

R = slope of the high fluence region of the curve

τ = measure of the zero swelling intercept of the extrapolated high fluence region

α = measure of the rate of transition from the onset of swelling to the high fluence stage of the curve.

TABLE HEDL.18

SWELLING IN S.T. TYPE 316 STAINLESS STEEL*

Sample Source	$\phi t (10^{22} \text{ n/cm}^2)$	T(°C)	$\Delta V/V_0$
Subassembly X041	2.6	817	-0.05
Canel Heat	2.5	817	-0.06
Heat #332990	2.3	817	-0.02
	1.9	817	-0.01
Pins B24, B25 & B27 HEDL	1.83	380	0.27
	2.35	440	0.33
	2.9	650	-0.14
	3.05	790	-0.14
	3.05	850	-0.10
	2.99	740	0.04
	2.63	590	0.14
	2.07	465	0.26
	0.75	380	0.03
	0.96	430	0.08
	1.19	640	-0.12
	1.25	790	-0.15
	1.25	850	-0.13
	1.22	720	-0.11
	1.09	550	0.03
	0.85	460	0.03
	3.08	390	0.66
	3.96	465	0.73
	4.88	690	0.08
	5.14	815	-0.09
	5.14	870	-0.11
	5.04	780	-0.03
	4.47	610	0.09
	3.5	505	0.48
Subassembly X-057	5.1	799	0.55
G-Lot Tubing	5.1	727	0.14
Heat #332990	3.83	504	0.43
HEDL	3.83	688	-0.02
	3.83	677	0.09
	3.83	577	0.26
	1.28	710	-0.08
	2.28	782	0.03
	4.2	677	0.23
	4.2	682	-0.07
Subassembly X-021B	2.07	458	0.36
Canel Heat	2.75	456	0.71
HEDL	2.07	543	0.10
	2.75	543	0.11

* All data are HEDL except where indicated.

TABLE HEDL.18 (Continued)

Sample Source		$\phi t (10^{22} \text{ n/cm}^2)$	T(°C)	$\Delta V/V_0$
Subassembly X021C Canel Heat HEDL		1.27	471	0
		1.75	471	0.15
		2.50	533	0.27
		3.34	533	0.50
		7.16	758	-0.003
		6.30	758	-0.003
		5.85	758	-0.03
		4.57	758	0.02
		3.34	441	1.04
		2.50	441	0.53
		1.75	431	0.20
		1.25	431	0.21
		7.16	758	-0.03
		2.50	441	0.64
Subassembly X018 Canel Heat HEDL		2.7	610	0.29
		2.34	590	0.27
		1.89	560	0.33
		1.50	520	0.40
		1.14	480	0.33
		0.90	440	0.17
		0.25	370	0.01
		2.7	570	0.68
		2.34	550	0.64
		1.89	530	0.60
		1.50	490	0.51
		1.14	460	0.31
		0.90	430	0.20
		0.66	390	0.11
		0.25	370	-0.02
GE Fuel Pins	F2V	6.15	472	3.95
	F2V	7.03	494	7.48
	F2V	7.32	508	7.76
	F2V	7.32	522	8.7
	F2V	6.4	530	6.0
	F2V	5.28	530	3.0
	F2X	5.58	468	2.86
	F2X	6.56	512	5.86
	F2X	5.6	522	3.37
	F2H	3.85	512	1.41
	F2H	3.93	536	1.47
	F2H	3.77	542	1.38
	F2H	3.1	546	0.68
X034-A ORNL		1.9	450	0.13
		1.9	480	0.14
		2.3	580	0.16
		2.7	580	0.16
		2.3	630	0.04
		2.3	725	0.03

TABLE HEDL.19

SUPPLEMENTARY DATA ON SWELLING IN
NEUTRON IRRADIATED TYPE 316 STAINLESS STEEL
(Available as of 1973)

$\phi t, 10^{22}$ $n/cm^2 (E > 0.1 \text{ MeV})$	Temperature (°C)	% $\Delta V/V_c$	Comments
1.5	510	.06	TEM, ORNL
1.9	580	.05	TEM, ORNL
2.3	425	.2	AI (Bulk Density)
3	456	.3	TEM Fuel
3	489	.25	Pin, ORNL
3.5	520	.36	↓
3.5	557	.28	
3	533	.33	
3	563	.36	
1.3	413	.04	EBR-II Bulk
1.5	442	.2	Density Fuel
1.6	483	.07	Pins
1.5	523	.14	↓
1.3	555	.28	
0.8	574	.24	
2.5	406	.17	EBR-II Bulk
2.8	434	.29	Density Fuel
2.9	460	.55	Pins
2.9	488	.48	↓
2.8	511	.38	
2.5	532	.11	
2.1	565	.12	
3.1	401	.45	EBR-II Bulk
3.7	454	.94	Density Fuel
2.9	496	.25	Pins
3.1	407	.54	↓
3.7	485	.99	
2.8	555	.21	
4.4	405	.89	EBR-II Bulk
4.9	432	1.29	Density Fuel
5.1	471	2.4	Pins
4.7	504	1.75	↓
4.1	535	.65	
3.7	557	.19	
2.1	477	.25	HEDL-Pin B-40
2.3	477	.36	(Bulk Density)
2.4	649	.09	↓
2.3	421	.46	
1.9	421	.35	
1.5	421	.24

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FIGURE HEDL.84. Neutron Induced Swelling Data for Type 316 Stainless Steel (400-450°C).

HEDL 7310-76.10

FIGURE HEDL.85. Neutron Induced Swelling Data for Type 316 Stainless Steel (450-500°C).

FIGURE HEDL.86. Neutron Induced Swelling Data for Type 316 Stainless Steel (500-550°C).

FIGURE HEDL.87. Neutron Induced Swelling Data for Type 316 Stainless Steel (550-600°C).

Figure HEDL.88 shows the incubation parameter τ derived from analysis of the annealed 316 data, as well as lower bound values derived for the cold-worked 316 swelling data. It is obvious that at least part of the swelling suppression due to cold-work arises from an increase in the incubation fluence. The steady-state swelling rates deduced from this data set are shown in Figure HEDL.89 and approach 3% swelling per 10^{22} n/cm² ($E > 0.1$ MeV). This is higher than a previous estimate utilizing only the "unstressed" data of Table HEDL.18. The transition function α that was previously used for 20% CW 316⁽²⁾ was found to be adequate for the annealed data set and was retained.

The correlations chosen to represent these data are given below:

$$R = \exp (-49.8592 + 0.195283T - 1.87409 \times 10^{-4}T^2)$$

$$\tau = 1.0 / (7.98769 - 2.98448 \times 10^{-2}T + 2.87279 \times 10^{-5}T^2)$$

$$\alpha = 1.117 + 6.889 \times 10^{-3}T$$

It is necessary that the coefficients in the expressions for R and τ retain all significant figures as truncation will lead to substantial differences in low fluence swelling predictions.

Predictions of this correlation are shown in Figures HEDL.90 and .91 and indicate that very large swellings will be obtained with annealed 316 at typical fast reactor fluences. At low fluences a double peaked behavior is noted which is similar to many observations of double peaks in fuel pin profilometry measurements. The double peaks arise as a consequence of the maximum observed in the temperature dependence of the incubation parameter τ .

This correlation has several obvious deficiencies which are directly related to the fact that the data are obtained at relatively low fluences, and the highest swelling was observed in stressed fuel pin cladding. The effects of stress on swelling have not been experimentally determined but current theoretical estimates indicate that stress effects are negligible below 500°C but may lead to a 10-20% enhancement at 550°C. At higher temperature, theory indicates substantial enhancement of swelling for typical fuel pin cladding stress states and temperatures.⁽⁵⁻⁷⁾

Since the development of this correlation, limited new data have become available and are listed in Table HEDL.20. The X-157 data points are from unstressed material but the fluences are too low to place much confidence in the predictions. The X098 data were derived from profilometry measurements,⁽⁸⁾ with good agreement with the correlation above 550°C and no correlation at all below 550°C. Element D-5 was a mixed oxide fuel pin which was operated as a run-to-failure test. It had a complicated irradiation history, having a run at 45, 50 and 62.5 MW in various positions in row 4 and 5. Both the neutron flux profiles and temperature varied during its history. Since it exhibited ~27% peak swelling, however, an attempt was made to calculate lifetime average temperatures and flux profiles and use these to make comparison between the observed and predicted swelling. See Figures HEDL.92 and .93 for comparisons of data and predictions on X098 and D-5 fuel elements. The temperature and flux values chosen are shown in Table HEDL.20 although they may be revised at a later date. Although it is unrealistic to expect that we can model such a complicated history it is interesting that the magnitude of the swelling predicted is quite close to the observed swelling. Once again there appears to be some tendency to overpredict swelling below 500°C.

FIGURE HEDL.88. Values of $\tau(T)$ Derived for Type 316 Stainless Steel.

FIGURE HEDL.89. Steady-State Swelling Rates Derived for Annealed 316 Stainless Steel.

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FIGURE HEDL.90. Predictions of the Linear-After-Incubation Swelling Equation for Annealed 316 Stainless Steel.

PREDICTIONS OF SOLUTION-ANNEALED SWELLING EQUATION

FIGURE HEDL.91. Predictions of the Linear-After-Incubation Swelling Equation for Annealed 316 Stainless Steel.

FIGURE HEDL.92. Comparison of Data and Predictions for X098 Swelling Data.

FIGURE HEDL.93. Comparison of Data and Predictions for Swelling Data from D-5 Fuel Element.

TABLE HEDL.20

RECENT DATA ON SWELLING OF NEUTRON IRRADIATED TYPE 316 STAINLESS STEEL

Source	$\phi t/10^{22}$ n/cm ² (E>0.1 MeV)	Temperature (°C)	% $\Delta V/V_0$	
			Measured	Predicted
X-157 N-lot (Density)	2.14	498	.034	.014
	1.45	452	-.085	.040
X-098 (Profilometry)	8.8	609	4.3	4.3
	8.9	535	0.90	12.7
	8.6	454	1.5	6.9
	9.4	689	0.30	0.12
Nucem D-5 Fuel Pin Cladding (Density)	11.2	475	8.93	14.5
	12.2	500	15.9	20.7
	12.7	530	26.0	23.1
	13.0	535	26.4	23.6
	12.95	540	26.8	23.0
	12.90	543	27.2	22.5
	12.5	550	21.6	20.5
	12.3	555	23.8	19.1
	12.05	560	23.9	17.7
	11.8	565	24.8	16.1
	10.9	570	14.6	13.5
	9.6	590	6.33	7.9

We note in Table HEDL.18 that the high swelling data come from GE fuel pins and that the highest temperature in the fuel pin data set is 546°C. This means that stress effects on this correlation are not present above 550°C, where the correlation agrees with the X-094 data. The large overprediction below 550°C is therefore consistent with stress effects considerations.

3. Expected Achievements

A stress-free swelling correlation will be developed for annealed 316 stainless steel. The current stress-included data set will be reexamined as additional data become available, and corrections made when necessary.

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E. DOSE CONCEPTS FOR CHARGED PARTICLE SIMULATION EXPERIMENTS

F. A. Garner, G. L. Guthrie and T. K. Bierlein

1. Objective

The objective of this study is to provide an understanding of those factors which have an impact on the assignment of displacement doses associated with void growth induced in charged particle simulation experiments. The role of cascade size, surface sinks, dose-deposition gradients, momentum transport and material discontinuities associated with second phases and voids will be studied in this effort. The magnitude of uncertainties in displacement dose calculations will also be explored, and the results coupled to the HVEM-stereoscopic analysis of swelling profiles produced by nickel ion and electron irradiations.

2. Accomplishments and Status

a. Scope of Work. Previous reports⁽¹⁻³⁾ have concentrated on dose concepts for electron irradiations while the work of this reporting period is concerned with heavy ion irradiations. The majority of ion induced swelling data have been produced by thin-film sectioning techniques or step-height profilometry. Both of these techniques utilize standardized deposited dose calculations^{(4)*} and assume that the calculated displacement dose is the appropriate correlation parameter to be associated with a swelling value obtained at a particular depth. There is some concern, however, that the steep ion dose deposition gradients will be altered by point defect diffusion and surface proximity to yield an effective growth environment that exhibits a different behavior with depth. There are also calculational uncertainties associated with the nuclear energy transfer and range straggling calculations for heavy ions. Forward momentum transfer following the nuclear energy deposition event may also alter the depth dependence. Second phases and voids can add another time dependent distortion to the depth dependence of the effective dose concept required for correlation with void growth. Accordingly, then, this study is comprised of four sections:

- (a) depth dependent displacement curves,
- (b) uncertainties associated with these calculations,
- (c) perturbations on the calculations due to structural or compositional discontinuities,
- (d) influence of temperature on the establishment of an effective dose concept.

b. Review of Calculational Procedure. Participants in the Alloy Development Program have previously agreed to employ for dose calculations the recommendations of the working group on Displacement Models and Procedures for Damage Calculation.⁽¹⁾ In that document a secondary displacement model for heavy ions was proposed which assumes that the dose concept should be proportional to the initial number of displaced atoms created. The energy available to create these displacements was to be calculated using the energy partition theory of Lindhard, et al., and the computer code EDEP⁻¹ (to be described in Computer Physics Communications by I. Manning and G. P. Mueller) was recommended as a standardized tool for calculation of this quantity. Essentially equivalent computer codes have been developed by F. A. Garner of HEDL and Brice⁽⁵⁾ as well as J. H. Rosolowski of GE. All of these codes predict the most probable ion range R_p , the distribution of final rest positions for the ions expressed as a standard deviation ΔR_p , and a depth dependent nuclear energy deposition curve for either

*Also used in a study by I. Manning and G. P. Mueller, "Depth Distribution of Energy Deposition by Ion Bombardment," to be published in Computer Physics Communications.

the most probable ion or the full distribution of ions. The EDEP-1 and IONDOSE (developed by F. A. Garner of HEDL and soon to be published) codes utilize the LSS10 code of Johnson and Gibbons⁽⁶⁾ to provide the range, range statistics and energy deposition information. With the exception of the work of Brice, all codes utilize the KDL assumption⁽⁷⁾ that ions which have projected ranges other than R_p have an energy deposition behavior such that all ions have the same energy at any given fraction of their respective projected range. This requires that an ion of projected range $R_f \neq R_p$ have an energy deposition rate defined by the following relationship:

$$\frac{dE}{dX}(F) = \frac{R_p}{R_f} \frac{dE_p}{dx}(F)$$

where $F = X/R_f = X/R_p$.

The work of Brice treats the range and damage statistics in a more rigorous manner which leads to somewhat different values of ΔR_p .

Since the codes EDEP-1 and IONDOSE will be the major calculational tools in this effort, a comparison was made of the predictions of these codes and those developed independently of the LSS10 code. Comparisons of the various computer code predictions lead to the conclusion that all codes yield essentially equivalent results, with the exception that the work of Brice leads to a different straggling ΔR_p . Some of the many comparisons made are represented in Figures HEDL.94 through .96. Note in Figures HEDL.94 and .95 that when ΔR_p is adjusted to the value predicted by Brice, the predictions of the various codes tend to converge. This is an indication of the relative validity of the KDL assumption for this work.

Brice predicts that the standard deviation in ion rest positions, ΔR_p , is approximately 30-40% higher than that predicted by the standard calculational code EDEP-1, as shown in Figure HEDL.97. The net effect is to broaden the displacement peak and shift it toward the front surface slightly. In the region from which most specimens are extracted, this can lead to 10-20% difference in the calculated dose for 4-5 MeV nickel ions.

A comparison of the ΔR_p values calculated by J. Lindhard and by H. E. Schiøtt* shows that the EDEP-1 and IONDOSE predictions are consistent with the basic Lindhard theory as shown in Figure HEDL.98. This question will be explored in more depth in the next reporting period.

C. Defect Survivability in Displacement Cascades. Doran⁽⁸⁾ is currently investigating the effects of cascade energy and distribution on point defect survivability and effective dose concepts. Since the energy deposition characteristics of heavy ions change with depth, the mean primary knock-on energy distribution was investigated to determine the range of cascade sizes produced along the ion's projected path, and typical results are shown in Figure HEDL.99. Since the energy distribution is heavily weighted toward low energy transfers, much of the energy will be lost to subthreshold events resulting in no displacements. The mean knock-on energy is therefore quite sensitive to the displacement energy threshold chosen. The range 25 to 50 eV covers the most commonly assumed values of E_d . Although the mean energy transfer does not vary strongly with depth, it does vary more strongly than previously reported⁽⁶⁾ and is everywhere less than that expected in a neutron-induced event. Since Figure HEDL.99 depicts only the distribution for the most probable ion, it is expected that

*H. E. Schiøtt, University of Aarhus in Denmark.

FIGURE HEDL 94. Comparison Between Nuclear Energy Deposition Curves Generated by Rosolowski and Garner for Nickel Ion Bombardment of Zirconium.

FIGURE HEDL.95. Comparison of Various Nuclear Energy Deposition Curves Predicted for 316 Stainless Steel.

FIGURE HEDL.96. Comparison of Various Nuclear Energy Deposition Curves Predicted for 316 Stainless Steel. (Note that employing Brice's value of ΔR_p leads to reasonable agreement with IONDOSE predictions.)

FIGURE HEDL.97. Comparison of Rest Ion Range Statistics Predicted by Various Investigators. (Brice and Garner)

FIGURE HEDL.98. Comparison of Rest Ion Range Statistics Predicted by Various Investigators. (Garner and Schjøtt)

FIGURE HEDL.99. Prediction of Depth Dependence of Mean Primary Knock-on Energy for Copper Ion Irradiation of 316 Stainless Steel.

the distribution for all ions will be somewhat less steep. The distribution of knock-on energies about the mean value will be determined in a later reporting period.

d. Effect of Alloy Composition on Displacement Depth Profiles. In the Alloy Development Program a range of material composition in the Fe-Ni-Cr system will be studied using accelerator simulation techniques. The effect of composition on the displacement depth curve was studied and found to be quite significant as shown in Figure HEDL.100. For nickel ion bombardment, increasing nickel concentration shifts the peak to higher displacement levels and nearer to the specimen front surface. At all depths forward of the peak, pure nickel will suffer more displacements per incident ion than stainless steel and iron. This is due not only to the change in elemental identity but also to the change in density with composition.

FIGURE HEDL.100. The Predicted Depth Dependence of Nuclear Energy Transfer is Strongly Dependent on Elemental Composition.

A similar phenomena occurs in fast reactors for somewhat different reasons. Using the standardized displacement dose calculations⁽³⁾ for an average neutron at the core center of EBR-II leads to total displacement cross-sections of 445 barns for 18/10 stainless and 521 barns for pure nickel. The difference in reactor induced dose is somewhat smaller (~15%) than that expected from typical foil extraction depths for 5 MeV nickel ion irradiations (~35%).

Figure HEDL.100 shows that 316 stainless steel and iron have roughly the same displacement curve, and that Inconel X-750 and Inconel 600 are also roughly similar. The differences are not sufficient to justify separate curves for each alloy, within the accuracy of the calculations.

e. Effect of Temperature on Displacement Depth Profiles. Stereomicroscopy of the volume contained within the full ion range has been employed by Thomas⁽⁹⁾ to determine the density and size distribution of voids produced by a combination of ion and electron irradiation. Rowcliffe, et al.⁽¹⁰⁾ and W. G. Johnston of GE have also made full range determinations of ion induced swelling profiles with somewhat conflicting results. At issue here is whether the depth dependence of swelling should be directly proportional to that of the deposited dose calculations. Intuitively one would expect at higher temperatures that the driving force for void growth would be modified by point defect diffusion, smoothing out the steep gradients in deposited dose. Even if diffusion were absent altogether, however, there is no reason to assume that the swelling dependence should be linear with dose. At temperatures below the peak swelling temperature, recombination will be the dominant trapping mechanism, leading to point defect concentration profiles which approximate the dose to the 0.5 power. As the temperature increases, recombination trapping will diminish and physical sink trapping will become dominant, leading toward a nearly linear defect concentration with dose. The influence of diffusion will, of course, increase with temperature.

In order to study the effects of temperature on the development of an effective dose concept for ions, the computer program CREED⁽¹⁻³⁾ was modified to include a depth dependent displacement rate. CREED has been employed previously to study point defect concentration gradients generated near foil surfaces in electron irradiations. This work has shown that the parameter of interest is the growth fluid ξ defined as $D_V (C_V - C_V^*) - D_I C_I$. This term contains the diffusivities and concentrations of vacancies and interstitials, and defines the net flow of vacancies across the void surface. Since ξ depends not only on the radiation induced defect flow but on thermal reemission rates of vacancies, the equilibrium vacancy concentration at the void surface C_V^* is included. This leads to a growth fluid which is dependent on the void radius. The growth fluid profile with depth has been shown to be influenced by surface sinks to a much greater depth than are vacancy concentration profiles.⁽³⁾

The variables investigated to date have been the dislocation density, peak dose rate and temperature. Typical values chosen were $10^9 - 10^{11} \text{ cm}^{-2}$, 1×10^{-3} to $2 \times 10^{-2} \text{ dpa/sec}$ and 500-700°C, respectively. The model represented by these calculations is shown in Figure HEDL.101 where 5.0 MeV Ni^+ ions are incident normal to the front surface of a 316 stainless steel target. The peak damage rate occurs at approximately one micron and the rear surface is assumed to be at 2.5 μm , at which position the point defect gradients are required to be zero. The surface energy was assumed to be 1000 ergs/ cm^2 and the energy of self diffusion has been experimentally determined to be 4.65×10^{-12} ergs. The initial estimate of the vacancy formation energy was taken to be 2.2×10^{-12} ergs. The dislocation densities employed were assumed to be uniform throughout and the growth fluids were calculated for a test void whose diameter was 100Å. Otherwise no voids were included in this first stage of analysis.

For a low dislocation density of 10^9 cm^{-2} the growth fluid curve is distorted greatly by diffusion to the front surface and into the bulk material beyond the range. There is little resemblance of this profile to that of the ion damage profile. A typical profile is shown in Figure HEDL.102. Also shown in Figure HEDL.103 are the peak values of growth fluid for various temperatures and dose rates. Surprisingly, the depth dependence of these profiles exhibits the same shape at all temperatures and displacement rates investigated; only the

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FIGURE HEDL.101. Physical Model Represented in CREED Calculations.

FIGURE HEDL.102. Depth Dependence of Growth Fluid Profile at Low Dislocation Density.

FIGURE HEDL.103. The Temperature and Dose Rate Dependence of Peak Growth Fluid at Low Dislocation Density.

elevation and magnitude of the curves changed. At higher temperatures the growth fluid of a 100Å diameter void is partially or always negative, dependent on the generation rate.

At a dislocation density of $1 \times 10^{11} \text{ cm}^{-2}$ the growth fluid profiles tend to approach the shape of the dose profiles. The surface influence increases with temperature, manifesting itself in reduced or negative growth fluids near the front surface. The growth fluid peaks tend to become more steep with increasing temperature, as shown in Figure HEDL.104. Therefore, as the temperature increases, the depth dependence of the growth fluid is more sensitive to the shift from trapping by recombination to trapping by sinks than it is sensitive to the increase in diffusion. This is contrary to the intuitively expected result.

The decreasing influence of diffusion at higher dislocation densities is not unexpected however. The intervening sinks tend to screen the influence of the surface and unirradiated material beyond the range, thereby increasing the relative influence of the local source. This effect can be assessed in terms of relaxation distances for each parameter of interest. If there exists a local perturbation which changes C_V , C_I or ϵ , then the perturbation extends into surrounding regions. The $1/e$ relaxation length for this perturbation is χ where

$$\chi_{C_V} = (S + D_I C_I R)^{-1/2}$$

and

$$\chi_{\epsilon} = (S)^{-1/2}$$

R is the recombination coefficient and S is the trapping coefficient due to sinks other than

FIGURE HEDL.104. Depth Dependence of Growth Fluid Profiles at High Dislocation Density, as Sensed by a Void of 100Å Diameter.

recombination. S is defined by

$$S = \rho_D Z_V + 4\pi\rho_V R_V C,$$

The void and dislocation densities ρ_V and ρ_D represent the physical traps, where R_V is the void radius and C is a factor which represents competition effects between voids whose spheres of influence overlap. Z_V is the vacancy bias factor for dislocations.

For our current model, x_ξ reduces to an inverse square root dependence on ρ_D since no voids are assumed in this calculation. Therefore local perturbations are relaxed faster for higher dislocation densities. The impression implied by Figure HEDL.104 is somewhat misleading, however, since ρ_D is not constant with temperature and voids will also alter the profile. The full temperature dependence of growth fluid profiles must allow a temperature dependent ρ_D and ρ_V and also allow for depth dependence of these quantities.

3. Expected Achievements in the Next Reporting Period

The expected temperature dependence of swelling profiles will be investigated further as well as perturbations to the depth dependence of dose calculations that arise due to voids and second phases.

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F. DEVELOPMENT OF AN ELECTRON BEAM WELDING PROCESS FOR THE
REENCAPSULATION OF RADIOACTIVE SPECIMENS

R. R. Borisch, J. F. Bates, J. F. Kastl, T. K. Bierlein and D. C. Kaulitz

1. Objective

The design and fabrication of irradiation vehicles for experimentation in the EBR-II are essential to both the IDRM program and the Alloy Development Program. Successful completion of fabrication schedules often requires the timely implementation of innovative welding procedures. The inclusion of irradiated specimens in an experimental subassembly contributes greatly to the complexity of the welding procedures. The purpose of this work is to develop a welding procedure for the encapsulation of irradiated specimens into subcapsules comprising the second phase of the Metallurgical Variables experiment (MV-II).

2. Accomplishments and Status

a. Encapsulation of Metallurgical Variables II. A new technique for reencapsulation of preirradiated specimens was developed and used in the loading of nine in-core subcapsules contained in the three-capsule Metallurgical Variables experiment. The NaK loading and electron beam welding were accomplished without occupying a hot cell. This was done by use of a special purpose upright cask which was arranged to contain new subcapsules, initially loaded with irradiated specimens in-cell. The cask was designed with two top end plugs, a large one for shielding and a smaller one designed to fit inside the larger plug. The smaller plug is removable for NaK filling and gas purging operations. The cask containing the preloaded subcapsule was moved into a nitrogen filled glove box. After removing the top shielding plugs and inspecting the subcapsule by means of mirrors placed directly over the top of the subcapsule, the primary plug was replaced. A 12-inch long hypodermic needle was then connected to a 10 cc disposable syringe for NaK filling operations. After filling the syringe with the prescribed amount of NaK the needle was inserted through the smaller hole in the end plug. The tip of the needle was placed near the bottom of the subcapsule. A 90° bend in the needle allowed performance of this task with minimal personnel hand exposure. The NaK was then injected into the subcapsule and the hypodermic needle removed. This step of the operation is shown in Figure HEDL.105. The primary cask plug was again removed and the subcapsule reinspected via mirrors. If the NaK level appeared too high, due to a pocket of trapped gas, the hypodermic needle was carefully reinserted into the specimen area and moved around until the gas bubble was released. Caution was exercised during this part of the procedure to avoid touching the weld zone with the NaK contaminated needle. The subcapsule end cap was then fitted using a special tool built for that purpose. This tool consists of a 2-foot long piece of 1/2-inch diameter stainless steel tubing with a 90° bend in the center. One end was attached to a vacuum system and attached to the other end was a rubber washer sized to fit over the end cap. After the lid was placed on the subcapsule it was then secured by center punching. Again, radiation exposure was minimized by using pliers to hold the punch and by viewing the operation through a mirror. The primary plug was then replaced and the cask readied for transport into the electron beam welder. Immediately upon opening the glove box door, a needle was placed in the smaller hole and an argon cover gas applied to the subcapsule.

FIGURE HEDL.105. NaK Loading of Subcapsule Containing Preirradiated MV-II Specimens.

After insertion in the welder the large plug and argon line were removed and the weld chamber evacuated for ~30 minutes. The removal of the argon line is shown in Figure HEDL.106. The 300 pound cask could not easily be turned on a turntable in the welding chamber, so the electron beam itself was rotated using a commercially available device called a poly-pattern" generator. The welding procedures also had to be altered in order to permit operation with a distance of 20 inches between heat shield and joint. This distance is four times greater than normal.

After welding, the cask and subcapsule were moved to a cell capable of unloading the subcapsule. The subcapsules were collected at this point and shipped in a standard transfer cask to another cell where they were visually inspected (1X to 30X), recleaned, helium leak checked and loaded into standard B7A capsules. The B7A capsules were extended through the hot cell wall. Normal welding procedures could then be utilized in the final welding operations.

FIGURE HEDL.106. Preparation for Welding by Removal of Cover Gas Line.

3. Expected Achievements

In the future, this welding process will be employed to accomplish reirradiation of specimens from subassemblies X-233 (an advanced alloy irradiation experiment), and X-216 (a reference material irradiation experiment).

G. A RELATIONSHIP BETWEEN EQUIVALENT CHROMIUM CONTENT AND IRRADIATION INDUCED SWELLING IN 316 STAINLESS STEEL

J. F. Bates, G. L. Guthrie and T. K. Bierlein

1. Objective

The objective of this work is to obtain a consistent set of correlations between minor alloy variations and swelling behavior in 316 stainless steel. The development of such correlations will lead to rational modifications of compositional or processing specifications which will result in improved performance of the reference cladding and duct materials.

2. Accomplishments and Status

a. Introduction. It is well known that compositional variations can affect the irradiation induced swelling observed in the 300-series stainless steels. The difference in swelling between 304 and 316 has been reported⁽¹⁾ (cf. References 2 and 3). More recently, an investigation of the effects of minor alloy variations on swelling in 316 stainless steel has shown that variations in the concentration of several constituents such as Si, C, Mo and P can affect the swelling behavior of the material.⁽⁴⁾ In addition, titanium additions to 316 stainless steel have been shown to increase the swelling resistance of the alloy at various temperatures.⁽⁵⁾ Simulation experiments using 5 MeV ions⁽⁶⁾ have shown a strong compositional dependence on swelling (measured by the step-height technique) in a series of ternary Fe-Cr-Ni alloys. The purpose of the present report is to note a correlation between the α -stabilizing effect of alloy additions and the influence that the addition has on the irradiation induced swelling of 316 stainless steel. The correlation is described quantitatively in terms of an equivalent chromium content, using a modification of the concept of Pryce and Andrews.⁽⁷⁾

b. Experimental Considerations. The different alloys discussed in this report were produced as special vacuum melted heats based on a nominal AISI 316 composition. Detailed fabrication history, alloy composition, and swelling data are contained in Reference (4) and will not be repeated herein. Densities were measured by means of the Archimedeian technique, before and after irradiation.

All of the alloy specimens under discussion were water quenched from a 1050°C annealing temperature. The rapid quenching rate resulted in a room temperature structure similar to that observable at the annealing temperature. The subsequent reactor exposures were for short times and at relatively low temperatures (390°C - 465°C). It follows therefore that no significantly large fraction of the material would have undergone a phase change during the irradiation. Therefore, the applicable phase diagrams are discussed in light of those phase changes associated with composition changes at the annealing temperature. In utilizing binary phase diagrams in this interpretation the assumption is made that effects of additions of two different elements are simply additive rather than synergistic.

c. Results. The results will be reviewed for each element separately, finally being discussed in terms of an equivalent chromium content.

Silicon. The effect of silicon additions on the normalized (to a fluence of 1×10^{22} n/cm²) swelling in silicon modified stainless steel is shown in Figure HEDL.107. The major effect occurs in the composition range of zero to about 0.5 wt% silicon. Figure HEDL.108, an isotherm of the ternary iron-carbon-silicon phase diagram shows that at small weight percentages of carbon, the major effect of increasing silicon content at the 1050°C (1925°F) annealing temperature is to shift the equilibrium structure away from a single-phase, face-centered cubic γ region toward a two-phase region of α plus γ , the α phase being cubic body centered. The phase diagram shown in Figure HEDL.108 is an isotherm at 1000°C (1832°F), slightly lower than the temperature from which these alloys were quenched. The exact positions of the phase boundaries shift slightly at higher temperatures, but the overall effect of silicon additions, namely a tendency toward stabilizing the α structure, persists. The binary phase diagram of the iron-silicon system also shows that the addition of a few percent silicon and subsequent heat treatment in the range 1040°C (1900°F) to 1150°C (2100°F) tends

FIGURE HEDL.107. Irradiation Induced Swelling in Silicon Modified Stainless Steel.

FIGURE HEDL.108. Fe-C-Si System. Isotherm at 1000°C (1832°F) References 13-16.

towards the formation of a two-phase ($\alpha+\gamma$) region. In the data set cited, silicon additions promote the transition to a body centered cubic structure and simultaneously reduce the swelling.

Phosphorus. Swelling as a function of phosphorus content at a fluence of around $2.4 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) is shown in Figure HEDL.109. As shown, even slight increases in

FIGURE HEDL.109. Swelling in Phosphorus Modified Stainless Steels at a Fluence of $2.2\text{-}2.6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$)

in phosphorus content, for example, from 0.01 to 0.04 wt% phosphorus, result in a reduction of swelling on the order of about a factor of two. Figure HEDL.110, a portion of the binary phase diagram for the iron-phosphorus system, shows that at approximately 1040°C (1900°F), the boundary between a two-phase region of the γ plus α and a single-phase γ region occurs at about 0.2% phosphorus. Thus, a correlation may be noted here also, namely, slight amounts of phosphorus reduce the swelling, and also result in a structure tending toward a transition from γ to α .

Molybdenum. As shown in Figure HEDL.111, the addition of molybdenum to 316 stainless steel also tends to reduce the irradiation induced swelling. Consider concentrations of molybdenum from 0.01 wt% to approximately 5 wt% in light of the phase diagram shown in Figure HEDL.112. Molybdenum additions stabilize certain phases in the carbon-iron-molybdenum system. At approximately 0.05% carbon a transition to a two-phase region occurs when the molybdenum content exceeds $\sim 2.4\%$. The difference in swelling between 304 and 316 stainless steel can be rationalized on the basis that 304, with no molybdenum, exists as a single-phase

FIGURE HEDL.110. Partial Region of the Fe-P System from Reference 10.

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FIGURE HEDL.111. Swelling in Mo-Modified 316 Stainless Steel.

FIGURE HEDL.112. Section of Fe-C-Mo System, Isotherm at 1000°C (1832°F).
References 11 and 12.

γ structure, and therefore is less swelling resistant than the same steel with 2.3% Mo. The addition of molybdenum tends toward a transition to a two-phase α plus γ region and provides increased swelling resistance.

Copper. Increasing the copper content (copper levels investigated ranged up to approximately 5 wt% copper) was ineffective in improving swelling resistance. Consideration of the copper-iron phase diagram shows that additions up to 5 wt% copper do not invoke a tendency toward transition to an α , or the body centered cubic structure, as has been the case in the previous examples, but a tendency toward a face centered cubic, two-phase region of γ plus ϵ . Thus, in the absence of a tendency toward a transition to a body centered cubic structure, no increase in swelling resistance is observed.

d. Discussion. The effect of an alloy addition on the irradiation induced swelling of 316 stainless steel appears to be consistent with the tendency of the addition to produce a transition from an FCC γ region to a BCC α region. In the examples discussed above, when such a tendency was established, the swelling was reduced. It is important to note that the swelling data cited above are for relatively low fluences, and that if the rate theory of swelling (i.e., an incubation period followed by a steady-state region of swelling) is applied, we must conclude that the data apply mainly to the initial incubation stage rather than the steady-state swelling region. The effects therefore pertain only to the as-heat-treated structure, and not to a steady-state swelling region after multiple changes in phase, precipitation, etc., have been effected. This concept would suggest that the temperature of the preirradiation annealing treatment could affect swelling in an annealed material. The temperature and cooling rate would dictate the initial starting structure, its relationship to the two-phase region, and thus the initial swelling.

Carbon concentration was the same ($\sim 0.05\%$) for all of the alloys discussed in this report. However, carbon is known to alter the swelling in 316 stainless steel^(4,8) and variations in

dissolved carbon by means of preirradiation heat treatment could also have a major influence on swelling.

It is expedient at this time to consider an empirical method of representing this observation of reduced swelling due to α -stabilization. One such method is the use of equivalent chromium content as a measure of the tendency to stabilize the α -phase. Pryce and Andrews⁽⁷⁾ have suggested a representation of this equivalent chromium content which we apply with an additional empirically derived term to account for phosphorus additions:

$$Cr' = Cr + 3Si + F Mo + 150P \quad (1)$$

where the element constituents are given in weight percent, and

$$F = 3 \tanh^8 (Mo) + 1 \quad (2)$$

Equation (2) allows the potency of Mo to vary from ~ 1 at Mo contents of $\sim 0.5 - 1.0\%$ up to ~ 4 at Mo contents of $\sim 3\%$. This variance in Mo factor is consistent with the observations of Pryce and Andrews which we have attempted to put into analytical form utilizing the tanh function.

The elemental additions which appear to have the most pronounced influence on swelling in 316 stainless steel are all α -stabilizers. It is, therefore, implicit in our discussion that the equivalent nickel content [which can be determined utilizing a formulation similar to Equation (1)] of the alloy remains constant.

This equation has been applied to the data with the results tabulated in Table HEDL.21 and plotted in Figure HEDL.113. It can be seen that there is a good correlation between the observed swelling normalized to fluence and the equivalent chromium content for a given alloy.

TABLE HEDL.21
ALLOY COMPOSITION AND SWELLING DATA

Alloy	Cr	Si	Mo	P	Cr'	$\% \frac{\Delta V}{V}$	ϕt^*	$(\% \frac{\Delta V}{V})/(\phi t)$
6	16.89	.35	2.36	.001	26.59	.31	2.20	.14
12	17.01	.36	2.33	.039	32.28	.037	2.27	.016
25	16.88	.01	2.31	.010	26.64	.19	1.96	.097
26	17.00	.48	2.34	.009	28.18	.054	1.97	.027
30	16.89	.40	.010	.010	19.50	.28	1.53	.18
31	16.86	.38	.11	.010	19.61	.28	1.53	.18
32	16.90	.40	1.00	.010	20.94	.12	1.53	.078
33	16.96	.38	3.03	.011	31.52	.034	1.53	.022
34	17.09	.40	4.93	.011	39.65	.018	1.53	.012

*Units of 1×10^{22} n/cm² (E>0.1 MeV)

This correlation can be employed to evaluate the relative swelling behavior of various heats of 316 stainless steel. The criteria which must be applied in such an evaluation are that the carbon content and equivalent nickel content of an alloy must remain relatively constant.

FIGURE HEDL.113. Swelling vs. Equivalent Chromium Content in 316 Stainless Steel

It is not the intent of this report to expound on the fundamental mechanisms which may be responsible for the correlation observed. There are two concurrent changes in the alloy system, however, which are worthy of mention. The first is the free energy changes associated with the transition to a two-phase region. It would appear that all of the α -stabilizers of steel are effective in reducing the swelling but at this time a generalization such as this may be premature. In dealing with the nucleation phase of swelling, we are dealing with a localized phenomenon. Russell⁽⁹⁾ has treated several of the possible effects of impurity additions on swelling. However, the effects are regulated by such a large quantity of variables that it is impossible at this time to conclusively decide which ones are dominating.

The second effect of interest comes from consideration of the work conducted by W. Hume-Rothery.⁽¹⁷⁾ That is, as these α -stabilizers are added, the electron concentration is increased. The influence of this effect on the formation of void nuclei is quite uncertain as local electronic configurations are probably extensively perturbed by the presence of interstitials and vacancy supersaturations.

One method of analyzing for effects of electron perturbations is to consider average electron density (or electron "hole" variations as they are sometimes referred to). Both initial^(18,19,20) and more recent⁽²¹⁾ treatments of this effect do not incorporate variations of silicon and phosphorus into the calculation. However, increases in molybdenum have

been shown to increase the average electron hole number, N_V . This would suggest that as N_V increases swelling may decrease.

The above technique has been applied to commercial heats with remarkable success as shown below:

<u>Heat Identification</u>	<u>Swelling Resistance Based on Data</u>	<u>Calculated Swelling Resistance Based on Chromium Equivalent Analysis</u>
DO Heat	Highest	Highest
GE Heat(M2783)	2	2
Program Heat (65808)	3	4
AI Heat(34357)	4	3
N Lot (87210)	Lowest	Lowest

The only discrepancy is the difference between the rankings of the Program and AI heats. Consideration of the carbon levels shows that the carbon is substantially lower in the AI heat than in the Program heat. We therefore conclude (based on the Met. Variables swelling data for carbon modifications) that the AI heats should indeed swell more than the Program heat. This does not invalidate the Chromium Equivalent calculations as carbon content is not yet directly accounted for in this technique.

e. Conclusions. In summary, examples are cited which show that several alloy additions which reduce the neutron-irradiation-induced swelling of 316 stainless steel are also α -stabilizers. This dependency of swelling on the initial crystal structure can explain why preirradiation annealing temperature of the alloy influences the resultant swelling. Application of this technique can yield valuable insight into the relative swelling behavior of different heats of 316 stainless steel for LMFBR applications.

3. Expected Achievements

Investigations relating to phase stability and irradiation induced swelling will be continued during the next reporting period. In addition, the concept of equivalent chromium content will be utilized in the design of a third generation Metallurgical Variables experiment (MV-III).

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H. EXPERIMENTAL EVIDENCE FOR STRESS ENHANCED SWELLING

J. F. Bates, E. R. Gilbert and T. K. Bierlein

1. Introduction

Radiation induced void formation or swelling in reactor core structural materials has been recognized as being an important phenomenon and one which must be considered in the design of LMFBRs. Experimental and theoretical studies have been conducted to quantify the swelling behavior of metals and alloys. The most abundant source of specimens for swelling analyses has been reactor core components which had received high exposure to radiation and therefore display measurable levels of swelling.

Theoretical studies have predicted that the hydrostatic stress in a reactor core component may affect the swelling rate. Because the reactor components from which most high fluence swelling data have been obtained are not free of stresses, there remains the possibility that the observed swelling behavior reflects some effect of stress. Unfortunately, the stress histories of such reactor components from which swelling data have been extracted have not been sufficiently well known to allow a clear assessment of the effect of stress on swelling.

An experiment was therefore devised to obtain swelling data under conditions where the stress is known. Possible effects on swelling due to chemical and mechanical interactions between the structural material and fissile material, such as prevail in fuel pins, were avoided by irradiating internally pressurized tubing. Some initial results of such an experiment are described in this report.

2. Experimental Details

This experiment, called "Stress Effects on Swelling," utilized a series of B-7a type capsules which were irradiated in EBR-II in subassembly X-157. The specimens consisted of pressurized tube segments manufactured from HEDL heat #V87210 in both the 20% cold-worked condition and the annealed (1010°C/6 minutes/air cooled) condition. The specimens were irradiated in thick walled pressure-pulse protective subcapsules and were immersed in static sodium. Helium was used as a pressurizing medium.

Since the cold-worked tubes showed no swelling at a fluence of $\sim 3 \times 10^{22}$ n/cm² (E>0.1 MeV), this report will be limited to describing the solution annealed specimens which were irradiated at two different temperatures: 430 and 500°C. These temperatures are based on heat transfer calculations and are estimated to be accurate to within $\pm 20^\circ\text{C}$. Fluctuations in temperature up to $\pm 10^\circ\text{C}$ probably occurred due to changes in local reactor power during the period of residence for this experiment. Following irradiation, the diameters of the specimens were measured to determine the creep deformation. The end caps of the specimens were then removed and the densities of the cladding portion of the specimens were measured by Archimedian techniques to determine the swelling. A few selectively chosen specimens were analyzed for gas content to establish that the pressure had not decreased during the course of the irradiation. The irradiation schedules were to provide goal peak fluences of 0.3, 1.5, 3, 6, and 10×10^{22} n/cm² (E>0.1 MeV). The data described in this report refer to the first three fluence levels.

3. Results

The results of the stress effects on swelling experiment are tabulated in Table HEDL.22.

TABLE HEDL.22
ANNEALED 136 SS DENSITY MEASUREMENTS (SUBASSEMBLY X-157)

Hoop Stress (ksi)	Irradia- tion Temp. (°C)	r (cm)	z (cm)	X-157 (1553 MWd)		X-157A (6192 MWd)		X-157B (13275 MWd)	
				G _D † (dpa)	$\frac{\Delta\rho}{\rho_0}$ (%)	G _D † (dpa)	$\frac{\Delta\rho}{\rho_0}$ (%)	G _D † (dpa)	$\frac{\Delta\rho}{\rho_0}$ (%)
0	430	17.7	-23.6	.83	+.104	3.32	+.098	5.79	+.035
9.8	430	14.3	-23.6	.87	+.104	3.47	-.110	6.04	-.0739
15.4	450	14.3	27.2	.59	+.104	2.35	+.200	4.1	+.016
19.5	430	17.7	-23.6	.83	+.104	3.32	-.054	5.79	-.0589
29.3	430	17.7	-23.6	.83	+.015	3.32	+.160	5.79	+.070
58.5	430	15.1	-17.3	1.44	+.082	5.78	+.17	10.0	-.029
0	500	17.7	14.5	1.38	+.070	5.5	-.021	9.6	-.034
9.6	500	17.7	14.5	1.38	+.070	5.5	-.110	9.6	-.094
14.2	500	14.3	8.1	1.93	+.070	7.7	-.110	13.4	-.163
18.8	500	14.3	8.1	1.93	+.103	7.7	-.059	13.4	-.194
23.6	500	14.3	8.1	1.93	-.013	7.7	+.18	13.4	-.187

Although some trends can be noted, it should be pointed out that during a particular irradiation time, specimens were located at different elevations in EBR-II and consequently were irradiated to different neutron fluence levels. Nevertheless, comparison of the swelling values of specimens comprising the low, 0.3, the medium, 1.5, and the high, 3.0×10^{22} n/cm² (E>0.1 MeV), fluence capsules indicates that a linear dependence of swelling on atom displacements may represent the trends. Therefore, the data were normalized linearly to atom displacements of 1.38, 5.5, and 9.6 dpa. The normalized data for 500°C are shown in

Figure HEDL.114. Also shown on the figure is a functional representation which is developed below. Figure HEDL.114 clearly shows an effect of stress on swelling for the data set

FIGURE HEDL.114. Effect of Stress on Swelling in Annealed 316 Stainless Steel Pressurized Tubes.

normalized to an atom displacement value of 9.6 dpa. Some increase in swelling is indicated for the data set normalized to 5.5 dpa, and primarily data scatter are observed for the set normalized to 1.38 dpa.

The specimen with the highest stress in the 9.6 dpa set does not follow the trend established by the other four data points comprising the set; swelling is reduced rather than increased. At the intermediate displacement level, 5.5 dpa, the data in Figure HEDL.114 also show a maximum swelling value, at a stress just below the proportional elastic limit. The high stress point which is low on the highest dpa curve is also above the proportional elastic limit of 15 to 19 ksi for this lot of annealed 316 stainless steel.

The swelling in the specimens irradiated at 430°C was much less and in fact not significantly above the level of data scatter. Higher fluence data will be required to perform an analysis.

4. Discussion

This is the first set of data which unambiguously shows an effect of stress on swelling. The most convincing set of data is at the highest fluence where the swelling is above the level of data scatter. Several important observations can be derived from the experimental

data. First, the data plot in Figure HEDL.114 shows that the enhanced swelling is linear in stress at low stress levels. This leads to the formulation:

$$S_T = S_0 (1 + P\sigma), \quad (1)$$

where S_T is the total swelling, S_0 is the stress-free swelling, and P is a coefficient. Secondly, if the value of P derived from the highest fluence level is compared with the average value of P computed for the intermediate fluence level, one concludes that P is independent of fluence. Comparisons between different sets of data points for different fluence levels can be used to evaluate P . When this is done, the value of P is computed to equal about 0.17 ksi^{-1} .

At stress levels above the proportional elastic limit, the effect of stress on swelling appears to be the reverse of that observed below the proportional elastic limit. It is well established that cold-work reduces swelling,⁽¹⁾ and it appears that the increasing amount of cold-work developed by stressing the specimens above the proportional elastic limit causes a reversal in the apparent effect of stress on swelling. The stress-strain relationship for the diametral strain during preirradiation pressurization of these specimens at temperatures is shown in Figure HEDL.115. The effect of cold-work upon swelling in this same heat of 316 stainless steel is shown in Figure HEDL-116.

FIGURE HEDL.115. Stress-Strain Curves for FTR Prototype Annealed 316 Stainless Steel Pressurized N-Lot Tubing at 825 and 1000°F.

FIGURE HEDL.116. Effect of Cold-Work on Swelling in 316 Stainless Steel (constructed from data of Reference 1).

In this discussion, strain and reduction of area of tubing are used interchangeably. This is a good approximation for the small strains discussed in this report, which are all less than 10%.

Indeed the data point corresponding to the highest stress level in the 9.6 dpa data set presented in Figure HEDL.114 can be adjusted for the effect of "cold-work" (increased dislocation density) due to straining. If this is performed from information provided in Figures HEDL.115 and .116, then the data point in Figure HEDL.114 can be raised up to the linear trend line given by Equation (1).

The primary effect of cold-work upon swelling reduction has been attributed to the dislocations generated by cold-working. The presentation which follows is an attempt to describe why increasing stress causes swelling to increase up to the proportional elastic limit and then to decrease as dislocations are generated when the proportional elastic limit is exceeded.

The total swelling in a material subjected to an applied stress σ which results in the generation of a dislocation density ρ_T , if straining occurs, may be represented by the following equation:

$$S_T = F(\phi, T, \sigma, \rho_T), \quad (2)$$

where F is a function of ϕ , the neutron fluence, T the temperature, σ the stress, and ρ_T the dislocation density. The total differential of Equation (2) is:

$$dS_T = \frac{\partial F}{\partial \phi} d\phi + \frac{\partial F}{\partial T} dT + \frac{\partial F}{\partial \sigma} d\sigma + \frac{\partial F}{\partial \rho_T} d\rho_T. \quad (3)$$

The first two terms of Equation (3) represent the stress-free swelling component and can be represented by the term dS_0 . The derivative of S_0 with respect to stress is zero. The derivative of S_T with respect to stress is then:

$$\frac{dS_T}{d\sigma} = \frac{\partial F}{\partial \sigma} + \left(\frac{\partial F}{\partial \rho_T} \right) \left(\frac{d\rho_T}{d\sigma} \right). \quad (4)$$

The first term which represents the partial derivative of swelling with respect to stress has been discussed elsewhere^(2,3) and is generally treated as being equal to a coefficient, P , times the stress-free swelling component, S_0 . This definition is compatible with Equation (1).

The two factors comprising the second term in Equation (4) represent the effects of an applied stress on swelling brought about by a change in the dislocation density. The first of these components, the partial derivative of F with respect to ρ_t , can be evaluated by two different means, one of which has been discussed above.

The change in swelling with respect to this increased dislocation density can be evaluated with data from other sources.^(1,4) Data from Reference 1 relate swelling to percent cold-working at 420°C. Reference 4 provides data showing the increase in dislocation densities with percent cold-working, as shown in Figure HEDL.117. Defining ρ_0 to be the dislocation density of an "annealed" specimen and utilizing these concepts, an equation of the following type can be used to describe the data in References 1 and 4.

$$\left(\frac{S_T}{S_0} \right) = K \left(\frac{\rho_0}{\rho_t} \right)^C. \quad (5)$$

When this is done, the correlation coefficient, 0.99, indicates a near perfect fit with the following values for the coefficients: $K = .95$, $C = 0.49$. Thus the ratio of the swelling in a strained specimen to the swelling in an annealed specimen is inversely proportional to the square root of the dislocation densities in these samples.

The change in dislocation density with respect to stress has been determined for Type 316 SS⁽⁵⁾ to be the form:

$$\rho = \alpha \sigma^2. \quad (6)$$

For the stresses involved in this experiment, Equation (6) is in agreement with the result of investigations for other metals.^(6,7) Substituting Equations (5) and (6) into Equation (4) and integrating from the initial swelling of S_0 , and an applied stress = 0 to a final swelling S_t at an applied stress = σ , results in the following equation:

$$S_T = S_0 \left[1 + P \left(\sigma - \beta \ln \sigma / \sigma_p \right) + \beta \left(\frac{1}{\sigma} - \frac{1}{\sigma_p} \right) \right], \quad (7)$$

FIGURE HEDL.117. Dislocation Density vs. Percent Cold-Work in 316 Stainless Steel.

where σ_p is the maximum swelling stress. It is felt that σ_p is associated with the unirradiated proportional elastic limit of the material.

At low fluences, where swelling is first detectable, this swelling peak appears to be consistent with the PEL. However, at higher fluences sufficient dislocation recovery reduces the effectiveness of the cold-worked structure and the swelling peak appears to shift to higher stress levels. The present data show peak swelling at stresses of 15 and 19 for exposures of 5.5 and 9.6 dpa, respectively. These values were used in Equation (7) (the predicted stress effect is shown in Figure HEDL.114. As higher fluence data become available, σ_p can be determined as a function of fluence.

Equation (7) represents the sum of two competing terms: a stress-assisted growth term tending to increase the swelling, and a second term tending to decrease the swelling due to an increased sink density, resulting in much the same reduction in swelling as by prior cold-working.

Figure HEDL.114 shows this equation applied to data at 500°C, for values of $P = 0.17 \text{ ksi}^{-1}$ and $\beta = 50 \text{ ksi}$ (note that when $\sigma < \sigma_p$, $\beta = 0$). equation is governed by two major items:

the initial, or stress-free, swelling and σ_p , the stress at which plastic yielding begins. The validity of the formulation will only be as good as those values of S_0 and σ_p which are determined. In the equation which we have developed, we apply no correction for thermal densification. An implicit assumption is that the thermal densification will manifest itself in variations in the S_0 term and that these thermal densifications will be relatively independent of the applied stress. This assumption is currently being investigated and will be reported at a later date. Until such a formulation is complete, it is felt Equation (7) should not be used for $S_T < 0$.

5. Summary

In summary, experimental evidence has shown that an applied stress increases the irradiation induced swelling in annealed AISI Type 316 stainless steel. The amount of this increase is linear (at low stress levels) with stress and reaches a maximum value at a stress just below that at which plastic yielding begins. The swelling then decreases nonlinearly with increasing stress until it eventually reaches the amount of swelling for unstressed material.

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