IN-REACTOR DEFORMATION AND FRACTURE OF
AUSTENITIC STAINLESS STEELS*

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ABSTRACT

An experimental technique for determining in-reactor fracture strain was developed and demonstrated. Differential swelling between a sample holder and a test specimen with a lower swelling rate produced uniaxial deformation. In-reactor deformations of 0.7 to 2.1% were achieved in type 304 stainless steel previously irradiated to fluences up to 8.8×10^{26} n/m² without fracture. These strains are significantly higher than found in postirradiation creep-rupture tests on similar samples. From the measured strain values and published irradiation creep data and correlations, the stress levels during the irradiation were calculated. On the basis of previous postirradiation creep-rupture results, many of the samples that did not fail would be predicted to fail. Thus we conclude that the in-reactor rupture life is longer than predicted by postirradiation tests.

Strain in a fractured sample was estimated to be less than 3.8%, and the in-reactor fractures were intergranular — the same fracture mode as found in postirradiation tests. Irradiation creep may relax stresses at crack tips and sliding boundaries, thus retarding the initiation and/or growth of cracks and leading to longer rupture lives in-reactor. However, the very high ductility or superplastic behavior predicted by the strain rate sensitivity of irradiation creep is not achieved because of the eventual interruption of the deformation process by grain boundary fracture.

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INTRODUCTION

At temperatures below approximately $T_m/2$ (T_m is the melting temperature in K) the strain rate resulting from irradiation creep processes can be several orders of magnitude greater than that

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attributable to thermal creep.^{1,2} Irradiation creep is a weaker function of temperature than thermal creep, and the strain rate depends on a low power of stress (i.e., creep rate varies nearly linearly with stress). Thus, the strain rate sensitivity, m ($m = \partial \ln \sigma / \partial \ln \dot{\epsilon}$), is large. Large values of m are required for and associated with superplastic behavior. Nichols³ predicted greatly enhanced ductility, even superplastic behavior for irradiation creep of Zircaloy because of the observed large m value and the lack of "embrittling" effects as evidenced by a large reduction of area. During irradiation, some examples occur of larger amounts of deformation than would be expected on the basis of postirradiation tests. However, there is essentially no information on the fracture mechanisms, ductility limits, and rupture times during irradiation and how these properties depend on strain rate, stress state, irradiation temperature, and neutron flux.

The present work was undertaken to (1) develop a technique for determining ductility limits and for investigating the fracture mechanisms during irradiation, (2) investigate the behavior of previously irradiated type 304 stainless steel, and (3) make some limited measurements on the in-reactor deformation of 20%-cold-worked type 316 stainless steel.

EXPERIMENT DESCRIPTION

Uniaxial deformation of rod and sheet tensile samples was produced by specimen holders, which are shown schematically in Fig. 1. The body of the specimen holder was constructed of high-purity type 316 stainless steel (i.e., a Fe-17% Cr-14% Ni-2.5% Mo alloy with low C, S, P, Si, etc.)

¹D. R. Harries, *J. Nucl. Mater.* 65: 157-73 (1977).

²E. R. Gilbert and J. F. Bates, "Dependence of Irradiation Creep on Temperature and Atom Displacement in 20%-Cold-Worked Type 316 Stainless Steel," *J. Nucl. Mater.* 65: 204-09 (1977).

³F. A. Nichols, "Evidences for Enhanced Ductility During Irradiation Creep," *Mater. Sci. Eng.* 6: 167-75 (1970).

ORNL-DWG 75-10462R

(a)

ORNL-DWG 75-535R

(b)

Fig. 1. Schematic Drawings of Specimen Holders Used for In-Reactor Fracture Strain Measurements on (a) Rod and (b) Sheet Samples.

having a composition shown in Table 1. As reported by Leitnaker et al.,⁴ this alloy has a much shorter incubation period for the onset of swelling and a higher swelling rate than commercial type 316 stainless steel. For the presently described experiments a second melt of the alloy was produced. As will be shown this melt exhibited slightly different swelling behavior than reported by Leitnaker et al.⁴ (i.e., a higher swelling rate).

Table 1. Composition of High-Purity Type 316 Stainless Steel for In-Reactor Fracture Strain Specimen Holders

Element	Content (%)	Element	Content (%)
Cr	17.0	V	<0.001
Ni	16.7	Co	0.03
Mo	2.5	Zr	<0.001
Si	0.10	W	0.068
Mn	0.03	Cu	0.02
Ti	0.01	C	0.001
Al	0.02	N	0.004
Nb	0.02		

Sheet samples were machined from a type 304 stainless steel EBR-II safety rod thimble that had been previously irradiated to maximum fluences of about 9×10^{26} n/m². Four samples each having a gage section 3.18 by 1.02 by 15.88 mm were contained in each specimen holder. Compliance between the specimen and the holder was obtained by means of pins in the specimen shoulders. Irradiation temperatures of 400, 450, and 550°C were obtained by means of a gas gap between the specimen holder and the inside of the containment tube. Disks located on each end centered the holders in the containment tube.

Three rod specimens of 20%-cold-worked type 316 stainless steel were strained during irradiation like the type 304 stainless steel specimens described above. The assembly consisted of a rod specimen with threaded

⁴J. M. Leitnaker, E. E. Bloom, and J. O. Stiegler, "The Effect of Minor Constituents on Swelling in Stainless Steel," *J. Nucl. Mater.* 49: 57-66 (1973).

shoulders, a holder body, and threaded end fixtures as shown in Fig. 1. The specimen was assembled into the holder, and the end fixtures were tightened to remove all slack but not enough to apply a stress to the specimen. Keyways in the shoulders of the specimen and in the specimen holder prevented application of a torque to the specimen during assembly. This entire assembly was irradiated in a sodium-filled subcapsule. Irradiation temperatures of 450, 550, and 650°C were maintained by a gas gap between the surface of the subcapsule and the inside of the containment tube.

The samples were irradiated in EBR-II and received an exposure of 8980 MWd. The calculated centerline fluence was 2.5×10^{26} n/m² (>0.1 MeV). Uncertainties in the irradiation temperatures are estimated to be $400 \pm 10^\circ\text{C}$, $450 \pm 10^\circ\text{C}$, $550 \pm 15^\circ\text{C}$, and $650 \pm 25^\circ\text{C}$.

The total deformation accrued in each sample was determined by its length change. Irradiation creep strain (or plastic strain) was then determined by subtracting that contribution to length increase resulting from the assumed isotropic swelling. Fracture surfaces were examined by scanning electron microscopy.

RESULTS

Density decreases observed in the high-purity type 316 stainless steel specimen holders are given in Table 2. Data for irradiation temperatures in the range 450 to 600°C are plotted and compared with the results of Leitnaker et al. in Fig. 2. These two alloys having nominally the same composition clearly exhibit significantly different swelling rates and possibly different incubation fluences.

Table 3 summarizes the results for type 304 stainless steel samples that were fabricated from a previously irradiated EBR-II safety rod thimble. The neutron fluence, irradiation temperature, and density decrease during irradiation of the material as a safety rod thimble as well as the neutron fluence, irradiation temperature, total sample length change, and density decrease measured in the present experiment are given. Ideally, all samples in a given holder should exhibit the same total length change since they should be constrained to deform as

Table 3. Results of In-Reactor Swelling Driven Fracture Experiment on
Preirradiated Type 304 Stainless Steel

Holder	Neutron Fluence >0.1 MeV (n/m ²)	Irradiation Temperature (°C)	Sample	Previous Irradiation			Present Experiment			
				Fluence >0.1 MeV (n/m ²)	Irradiation Temperature (°C)	Density Change (%)	Length Change		Density Decrease (%)	ϵ_c Strain ^a (%)
							(mm)	(in.)		
X-6	1.3×10^{26}	400	Y-3-A	8.7×10^{26}	418	7.08	0.254	0.0100	2.28	-0.8
			Y-5-A	7.2	427	5.11	0.399	0.0157	2.17	0.2
			X-4-B	8.8	387	3.83	0.220	0.0087	1.96	-0.7
			Cont-2	0		0	0.104	0.0041	0.76	-0.1
Y-6	1.3	450	Y-14-B	2.1	465	0.30	0.454	0.0179	1.25	1.5
			B-21-B	1.1	370	0.24	0.360	0.0142	0.74	1.4
			X-6-A	7.5	377	2.98	0.447	0.0176	2.16	0.5
			Cont-8	0		0	0.261	0.0103	0.76	0.9
X-5	1.75	550	X-4-A	8.8	387	4.28	0.751	0.0296	2.74	1.8 ^b
			Y-14-A	2.1	465	0.30			1.46	Fracture ^c
			B-21-A	1.1	370	0.24			1.74	Fracture ^c
			Cont-1	0		0	0.490	0.0193	0.96	2.1
Y-5	1.75	550	X-6-B	7.5	377	2.98	0.858	0.0338	3.06	2.1
			Y-3-B	8.7	418	7.08	0.810	0.0319	3.52	1.4
			Y-5-B	7.2	427	5.11	0.861	0.0339	3.79	1.4

^a $\frac{(\Delta L)_{\text{Measured}} - (\Delta L)_{\text{Calculated}}}{\text{gage length}} = \text{Strain}$, where (ΔL) calculated is length change calculated from density change.

^b Fracture occurred in shoulder.

^c Fracture strain is calculated to be less than 3.7% by using length change of holder to estimate length change of sample.

the holder swells. Evidently the pins had deformed some since these length changes were not identical and the pins could not be forced from the holders during disassembly.

The strain values listed in Table 3 were calculated by subtracting the length increase attributable to the density decrease from the measured length increase and assuming that the resultant strain occurred over the gage section:

$$e_c = \frac{(\Delta L)_{\text{Total}} - \frac{1}{3} \frac{\Delta \rho}{\rho} L_s}{\text{gage length}} \quad (1)$$

where

$(\Delta L)_{\text{Total}}$ = measured specimen length increase,

$\left(\frac{\Delta \rho}{\rho}\right)$ = density decrease of entire sample, and

L_s = initial sample length.

This analysis would be in error if the stress present in the gage section affected the swelling. To investigate this question both shoulders were cut from four samples near the point where the gage section begins. Density was measured⁵ on the resulting three pieces from each sample. The resulting density profiles along the sample length are compared with the preirradiation profiles in Fig. 3. Recall that the density of each sample varied along its length before the in-reactor fracture experiment as a result of the temperature and fluence gradients along the EBR-II thimbles from which the samples were machined. Within experimental accuracy the density change in the gage section was the same as that in the specimen shoulders. The density decrease that occurred in the type 304 stainless steel samples during

⁵These measurements were made at Hanford Engineering Development Laboratory on equipment designed for density determinations on very small samples. The authors are indebted to J. J. Laidler and co-workers for their assistance.

Fig. 3. Postirradiation Density of Creep Samples. The density change in the shoulder was approximately the same as in the gage section. 1 in. = 25.4 mm.

the present irradiation was clearly a function of the prior history of the sample. This can be seen from Fig. 4, which shows the swelling rate observed in the present experiment as a function of the previous density change. This is an oversimplification of the situation since it does not take into account variations in the microstructure due to the previous irradiation history. However, larger previous density changes are clearly associated with higher swelling rates in the present experiment.

Fig. 4. Correlation Between Swelling Rate and Previous Density Decrease.

Calculated strain values are listed in Table 3. At 400°C the swelling rate in the high-purity type 316 stainless steel holders was near or slightly below that of the samples. This led to negative *calculated* strains by Eq. (1). At 450 and 550°C the specimen length increased more than could be accounted for by isotropic swelling. Samples that had not failed had strains in the range 0.7 to 2.4%.

Two samples had fractured. Both were contained in holder X5, which was irradiated at 550°C to $1.75 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$). The density decrease of these samples in the previous irradiation was small (Table 3), and thus the swelling rate in the present irradiation was low. The amount of plastic deformation or strain in these samples was thus larger than in other samples. Sample Y-14-A failed in the shoulder. Sample B-21-A failed in the gage, as shown in Fig. 5. By using the length change of the specimen holder to estimate total sample length change, the fracture strain is calculated to be less than 3.8%. In both samples the fractures were predominantly intergranular. The

(a)

(b)

Fig. 5. Fracture of Irradiated Specimens. (a) Sample Y-14-A, which fractured at the pinhole (approximately). (b) Sample B-21-A, which fractured in gage section (approximately). Scanning electron microscope examination showed the fracture to be intergranular.

entire fracture surface of sample B-21-A is shown in Fig. 6. At higher magnification (Fig. 7) oxide particles were clearly visible

Fig. 6. Fracture of Specimen B-21-A. Type 304 stainless steel irradiated at 370°C to 1.1×10^{26} n/m² (>0.1 MeV), then irradiated in stress effects experiment at 550°C to 1.75×10^{26} n/m² (>0.1 MeV). 40×.

Fig. 7. Oxide on Grain Boundary Fracture Surface of Specimen B-21-A from Stress Effects Experiment. Presence of oxide particles indicates that sample fractured at temperature during irradiation. 1000×.

on the grain boundary fracture surfaces, indicating that the fracture occurred while the sample was at temperature (i.e., during irradiation).

None of the three 20%-cold-worked type 316 stainless steel samples failed. Results of length and density measurements are listed in Table 4. Calculated strains vary with temperature (because of the temperature dependence of the swelling of the high-purity type 316 stainless steel holder material) and are in the range 2.4 to 5.6%.

Table 4. Results of Length Measurements on 20%-Cold-Worked Type 316 Stainless Steel Specimens Deformed In-Reactor

Sample	Irradiation Temperature (°C)	Neutron Fluence >0.1 MeV (n/m ²)	Length Increase, mm (in.)				Density Decrease ^a (%)	Strain ^b (%)
			Measured on Specimen		Calculated from Holder Density Change			
X-1	450	1.3 × 10 ²⁶	0.30	(0.012)	0.38	(0.015)	0.09	2.4
X-2	550	2.0	0.71	(0.028)	0.84	(0.033)	0.13	5.6
X-3	650	2.5	0.43	(0.017)	0.53	(0.021)	0.19	3.4

^a Measured on entire sample.

^b Assuming that the measured specimen length increase occurred in the specimen gage length.

ANALYSIS

Differential swelling produces internal stresses in a way similar to the production of thermal stresses. However, as swelling continues the stresses would build up indefinitely if they were not relaxed by irradiation creep and eventually by plastic flow. The highest level of stress produced depends on the balance between the swelling rate and the deformation rate. Under proper conditions, the stresses remain always below the yield point. For this case, an elementary analysis is developed and then applied to the analysis of the in-reactor fracture experiments.

For uniaxially loaded specimens, the total inelastic strain rate, $\dot{\epsilon}$, is given by

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

when thermal creep and plastic flow can be neglected. There, $\dot{S}$ is the swelling rate, B the irradiation creep coefficient in the absence of swelling, D is a swelling-related creep coefficient, $P\dot{S}$ is the stress-enhanced swelling rate, ϕ the neutron flux, and σ the uniaxial stress. The general form of the constitutive law, from which Eq. (2) is derived, was proposed earlier by Wolfer et al.⁶ and based on microstructural models for swelling and irradiation creep. Regardless of the validity of these models, one can combine all the stress relaxation mechanisms into one term $\psi\sigma$ by defining an irradiation creep compliance, ψ , and considering it as a material-dependent function.

In applying Eq. (2) to the swelling-driven fracture experiments we shall employ the subscripts h and s to distinguish between quantities for the holder and samples respectively.

If the ends of the holder are connected rigidly to the sample contained in it, then the total strain rates for holder and sample must be equal:

$$\dot{\epsilon}_s = \dot{\epsilon}_h . \quad (3)$$

Since the total strain rate is the sum of the elastic and inelastic strain rates, Eq. (3) gives

$$\dot{\sigma}_s/E_s + \dot{S}_s/3 + \psi_s \sigma_s = \dot{\sigma}_h/E_h + \dot{S}_h/3 + \psi_h \sigma_h . \quad (4)$$

We may now eliminate the stress in the holder, σ_h , by noting that the forces over the cross-sectional areas of holder and sample, A_h and A_s , must always balance. Hence

$$A_s \sigma_s + A_h \sigma_h = 0 , \quad (5)$$

⁶W. G. Wolfer, M. Ashkin, and A. Boltax, "Creep and Swelling Deformation in Structural Materials During Fast-Neutron Irradiation," pp. 233-58 in *Properties of Reactor Structural Alloys After Neutron or Particle Irradiation*, Am. Soc. Test. Mater. Spec. Tech. Publ. 570, American Society for Testing and Materials, Philadelphia, 1976.

and we obtain

$$\dot{\sigma}_s + \alpha \sigma_s = \beta, \quad (6)$$

where

$$\alpha = (\psi_s + \psi_h A_s / A_h) / (1/E_s + A_s / E_h A_h) \quad (7)$$

and

$$\beta = (\dot{\epsilon}_h - \dot{\epsilon}_s) / 3(1/E_s + A_s / E_h A_h). \quad (8)$$

A particularly simple and instructive solution of Eq. (6) is obtained for time-independent swelling and irradiation creep. In this case,

$$\sigma_s(t) = (\beta/\alpha)[1 - \exp(-\alpha t)]. \quad (9)$$

This solution shows that the stress builds up at an initial rate of β and approaches a saturation value of $\sigma_\infty = \beta/\alpha$. To evaluate Eq. (9) for the present experimental conditions, we note first that the ratio A_h/A_s is 10.8 and 17.5 for the rod and sheet samples, respectively. Therefore, we can neglect terms with A_s/A_h in Eqs. (7) and (8). Next, we use the parameters $\beta = 1.3 \times 10^{-30} \text{ m}^4/\text{Nn}$, $D = 4.84 \times 10^{-9} \text{ m}^2/\text{N}$, and $P = 0$, as reported by Flinn et al.⁷ for the irradiation creep of type 304L stainless steel. We find then that the time required to reach the value of $\sigma_\infty/2$ is about $5 \times 10^5 \text{ s}$, which corresponds to a fast neutron fluence of about 10^{25} n/m^2 . This time is short in comparison with the duration of the irradiation experiment reported in this paper, and we can, therefore, assume that each sample was subject to a saturation stress σ_∞ for the entire duration of the irradiation.

⁷J. E. Flinn, G. L. McVay, and L. C. Walters, *J. Nucl. Mater.* 65: 210-23 (1967).

In the case of the sheet samples, the pins that connected the holder and the sheet samples had also deformed. Therefore, Eq. (3) was not satisfied, and the saturation stress cannot be determined directly from Eq. (1). For this reason, the stresses in the samples were determined directly from the integrated form of Eq. (2); that is, from

$$\epsilon_c = \psi_s \sigma_s t, \quad (10)$$

where ϵ_c is the creep strain (total inelastic strain minus swelling strain) in the sample as listed in the last column of Tables 3 and 4. Using the measured irradiation creep coefficients for type 304L stainless steel⁷ and for 20%-cold-worked type 316 stainless steel⁸ we obtain the stresses listed in Table 5. They are in most cases sufficiently low to justify our assumption that ϵ_c is due to irradiation creep only. However, for the samples B-21-B and Cont-1 as well as for all the cold-worked samples the computed stresses are larger than yield strength, and we must conclude that plastic flow had occurred in these samples.

Let us now address the main question for this and future experiments, namely the embrittlement of irradiated materials tested in or out of reactor. Previous experiments yielded only postirradiation data on ductility and creep rupture, and these data are extensively used for in-reactor applications. When we compare the present in-reactor strains with postirradiation ductility data we find the former to be significantly larger. In fact, a postirradiation ductility of only 0.2% was found⁹ for type 304 stainless steel irradiated to a fast fluence of 6.5×10^{26} n/m² at 393°C and subsequently tested at 550°C. In contrast, the comparable in-reactor fracture test on sample X-4-A did not lead to failure even after a strain of 2%.

⁸E. R. Gilbert and J. F. Bates, "Dependence of Irradiation Creep on Temperature and Atom Displacements in 20%-Cold-Worked Type 316 Stainless Steel," *J. Nucl. Mater.* 65: 204-09 (1977).

⁹E. E. Bloom, "Irradiation Strengthening and Embrittlement," pp. 295-329 in *Radiation Damage in Metals*, American Society for Metals, Metals Park, Ohio, 1976.

Table 5. Stresses and Computed Rupture Times
for In-Reactor Fracture Samples

Holder	Sample	Stress ^a		Rupture Life, hr	
		(MPa)	(ksi)	t_r^*	t_r
X-6	Y-3-A	26	3.7	$>10^{16}$	$>10^{16}$
	Y-5-A				
	X-4-B				
	Cont-2				
Y-6	Y-14-B	323	46.8	2.3×10^4	8
	B-21-B	(476)	(69.1)		
	X-6-A	67	9.7	$>10^{11}$	$>10^{11}$
	Cont-8	186	26.9	1.2×10^5	23
X-5	X-4-A	186	26.9	4940	5
	Y-14-A				
	B-21-A				
	Cont-1	(528)	(76.6)		
Y-5	X-6-B	196	28.5	3100	2.4
	Y-3-B	109	15.8	6×10^5	2270
	Y-5-B	109	15.8	6×10^5	2270
	X-1	(3985)	(578)		
	X-2	(2965)	(430)		
	X-3	(600)	(87)		

^aValues in parentheses are stress levels above the yield stress. Plastic deformation is expected.

To explain this result one could simply argue that the contribution of thermal creep to the in-reactor strain is less than the postirradiation ductility value, and hence, no failure. The implicit assumption here is that irradiation creep has no effect on embrittlement. This interpretation, however, conflicts with the following creep-rupture considerations.

In Fig. 8 are shown creep-rupture data for unirradiated and irradiated type 304 stainless steel, plotted as a function of the Larson-Miller parameters. Data for the irradiated samples were obtained in post-irradiation tests.^{9,10} Utilizing these data and the stresses listed in

¹⁰E. E. Bloom and J. O. Stiegler, "Effect of Fast Neutron Irradiation on the Creep Rupture Properties of Type 304 Stainless Steel at 600°C," pp. 451-467 in *Irradiation Effects on Structural Alloys for Nuclear Reactor Applications*, Spec. Tech. Publ. 484, American Society for Testing and Materials, Philadelphia, March 1971.

Fig. 8. Larson-Miller Parameter Correlation for Type 304 Stainless Steel in Unirradiated Condition and After Irradiation to About $6 \times 10^{26} \text{ n/m}^2$. To obtain stresses in MPa, multiply by 6.895.

Table 5, we computed two rupture lives, t_r^* and t_r , for the in-reactor fracture samples, based on the creep rupture correlations for the unirradiated (t_r^*) and irradiated (t_r) cases. The computed rupture times are listed in Table 5, but only for samples that did not fracture or deform plastically. The predicted rupture lives should be compared with the irradiation time of 3500 hr. Based on correlations obtained from postirradiation creep-rupture tests, failure should have occurred for all samples except Y-5-A and X-6-A. Samples Y-3-B and Y-5-B would fall within the expected scatter of creep-rupture data (i.e., their predicted rupture life is close to the experiment duration). The fact that failure did not occur in a number of samples predicted to have very short rupture lives leads to the conclusion that the in-reactor rupture life cannot be predicted by and indeed is longer than that measured in postirradiation tests. However, the fracture mode is clearly intergranular for both in-reactor and postirradiation tests. For these irradiation and test conditions matrix strengthening and embrittlement of grain boundaries by helium appear to promote intergranular fracture⁹

and thus reduced ductility and rupture life. It has been suggested that strengthening of the matrix from irradiation damage (dislocation loops, network, voids, and precipitate particles) will reduce plastic flow at a crack tip or at points of constraint along a sliding grain boundary and produce increased stresses in such regions. The stress concentration will produce an increased growth rate for cavities near the crack tip relative to those removed from the crack. A crack will thus grow in a slow stable manner until reaching a critical size; there unstable growth will occur. The difference between stress-rupture life determined in-reactor and that determined in a postirradiation test may be associated with a reduction due to irradiation creep in the magnitude of the stress concentration that can be sustained near a crack tip.

As a result, the crack initiation and/or growth rate will be reduced and the rupture life increased. The grain boundaries clearly remain the weak link in the system. Very high ductility or superplastic behavior is not achieved because grain boundary fracture interrupts the deformation process.