ORNL-TM-3733

POSTIRRADIATION MECHANICAL PROPERTIES OF TYPES 304 AND 304 + 0.15% Ti STAINLESS STEEL

E. E. Bloom
J. O. Stiegler

OAK RIDGE NATIONAL LABORATORY
OPERATED BY UNION CARBIDE CORPORATION • FOR THE U.S. ATOMIC ENERGY COMMISSION

ORNL-TM-3733

Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

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Submitted to *Nuclear Technology*

JULY 1972

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U.S. ATOMIC ENERGY COMMISSION

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ABSTRACT

The tensile and creep-rupture properties of types 304 and 304 + 0.15% Ti stainless steels have been determined after irradiation at temperatures in the range 400 to 820°C to maximum neutron fluences of 3.5×10^{22} neutrons/cm^2 (> 0.1 MeV). Changes in mechanical properties were related to the microscopic observations of irradiation-produced defects.

When irradiated in the annealed condition in the neighborhood of 450°C, type 304 stainless steel exhibited an increased yield stress, reduced strain hardening coefficient, and reduced uniform and total elongation. The increased yield stress could be correlated with the strengthening expected from irradiation-produced voids and dislocations. With increasing irradiation temperature the concentration of these defects decreased and thus the magnitude of the yield stress increase became less. At 500 to about 600°C irradiation and test temperature a pronounced reduction in creep-rupture ductility was observed. Fractures were intergranular. It is suggested that in this temperature range the void-dislocation structure together with the transmutation-produced helium were responsible for the intergranular fractures and low ductilities. At higher temperatures no void-dislocation structures were formed. For these conditions the ductilities were higher than in the 500 to 600°C range but still significantly below the unirradiated value.

Variation in alloy composition and preirradiation microstructure had a strong influence on the postirradiation properties. Type 304 + 0.15% Ti stainless steel exhibited significantly higher tensile and creep-rupture ductilities than the standard alloy when irradiated and tested above 450°C. The strength properties of specimens irradiated in the 10% cold-worked condition were similar to those of material irradiated in the annealed condition, but the total elongation and reduction in area were slightly lower.

INTRODUCTION

Austenitic stainless steels have been selected for use as fuel cladding and structural components in Liquid Metal Fast Breeder Reactors. This choice was based upon high-temperature strength properties, resistance to corrosion by liquid sodium, compatibility with uranium-plutonium oxides, and the low neutron capture cross sections which are required for breeder reactors. Near-term reactors will use types 304 and 316 stainless steels. For reactor design it is necessary to define the effects of irradiation upon the physical and mechanical properties of these alloys. To increase the life of reactor core components and thus improve the economics of the system, it is desirable to optimize the resistance of these alloys to the various forms of radiation damage.

This report presents a summary of recent experiments in which the effects of fast-neutron irradiation on the microstructure, tensile properties, and creep-rupture properties of annealed type 304 stainless steel were investigated. Results of initial attempts to reduce the magnitude of the property changes in type 304 stainless steel through small adjustments in composition or preirradiation microstructure will also be discussed.

EXPERIMENTAL

Mechanical property test specimens with 0.125 in. diameter and 1-in.-long gage were irradiated in the reactor core in a row 2 position of the Experimental Breeder Reactor-II (EBR-II) to maximum fluences of 3.5×10^{22} neutrons/cm^2 (> 0.1 MeV). Specimens were irradiated at temperatures in the range 400 to 820°C. Irradiation temperatures above the ambient sodium coolant temperature were obtained by means of a gas gap between the surface of cylindrical specimen holders and the inside of the containment tube. Additional details of the experiment design have been given elsewhere.[1]

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[1] E. 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.

Microstructures of both as-irradiated and tested specimens were characterized by transmission electron microscopy. Dislocation loop, dislocation line, and void concentrations were determined from photomicrographs in which the foil thickness had been determined by stereomicroscopy. Tensile tests were conducted on Instron tensile testing machines in air at an initial strain rate of 0.002 min^{-1}. Creep-rupture tests were conducted in air in lever-arm creep machines. Specimen elongation as a function of time was determined by a linear differential transformer which measured the relative movement of the upper and lower specimen grips. Test temperatures were controlled to ±3°C. Specimens were tested at or near their respective irradiation temperatures.

RESULTS

Microstructures of Irradiated Materials

The microstructures present in specimens of type 304 stainless steel irradiated in the annealed condition (1 hr at 1050°C) are illustrated in Figs. 1 and 2. At irradiation temperatures of 590°C and lower, the microstructure contained voids, dislocation loops and lines, and a variety of precipitate particles. In general, concentrations of the defect structures decreased and average sizes increased with increasing irradiation temperature. Types and relative amounts of precipitate particles were also sensitive to irradiation temperature. At the lower irradiation temperatures (at these fluence levels) the voids appeared to be distributed uniformly throughout the matrix and not to be associated with any other structural features. There were, however, zones denuded of voids a few hundred angstroms wide adjacent to grain boundaries. No voids were detected in the grain boundaries. For irradiation temperatures in the range of about 500 to 600°C the voids were found in association with some needle-shaped precipitate particles [see Fig. 1(c)]. Brager et al.[2] have suggested that these are probably the Fe-Cr sigma phase, but analysis

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[2] H. Brager, J. L. Straalsund, J. J. Holmes, and J. F. Bates, *Met. Trans.* 2: 1893 (1971).

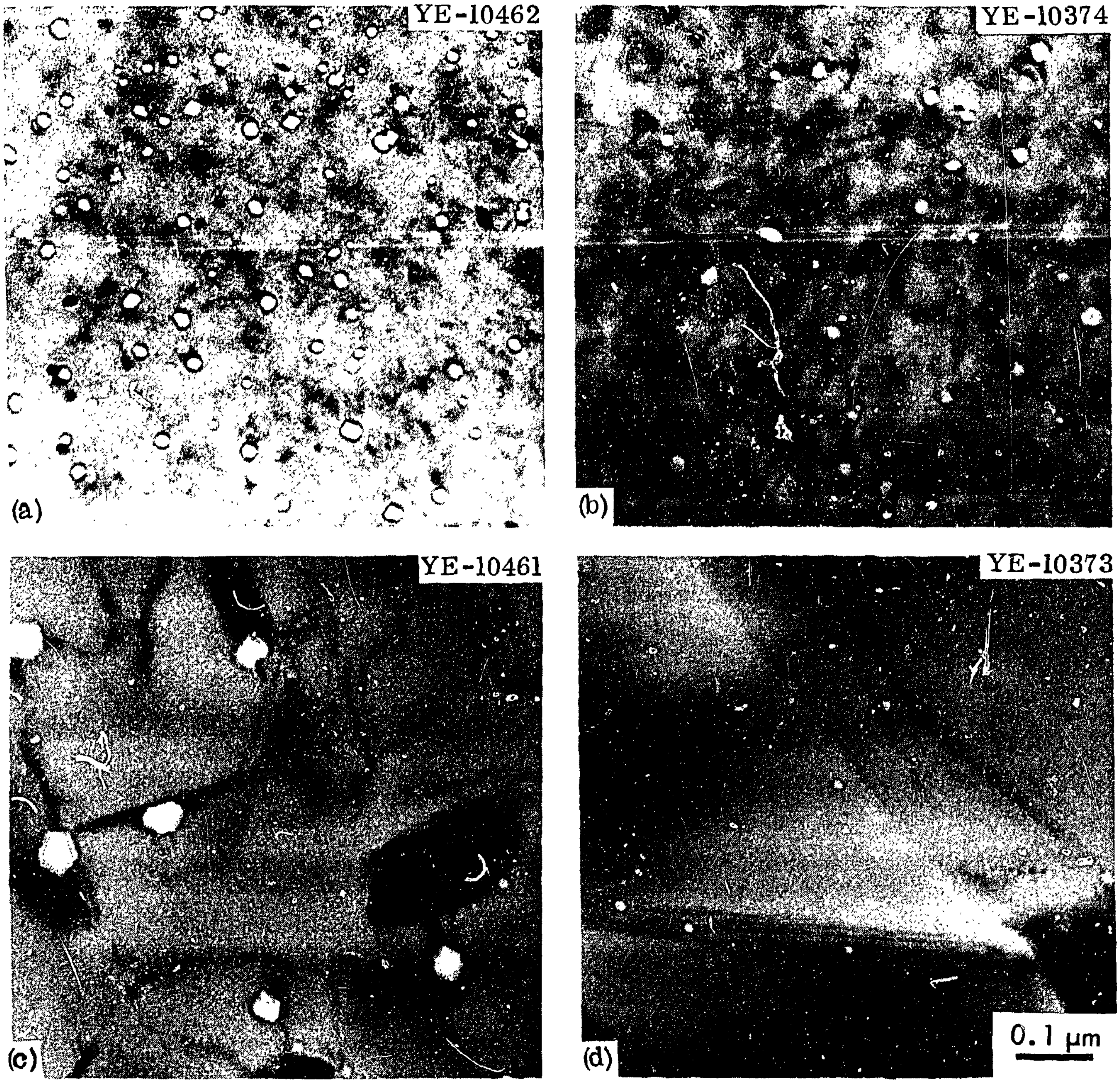

Fig. 1. Cavities Formed in Specimens of Annealed Type 304 Stainless Steel. (a) 440°C, 1.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV), (b) 535°C, 1.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV), (c) 590°C, 3.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV), and (d) 820°C, 3.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV).

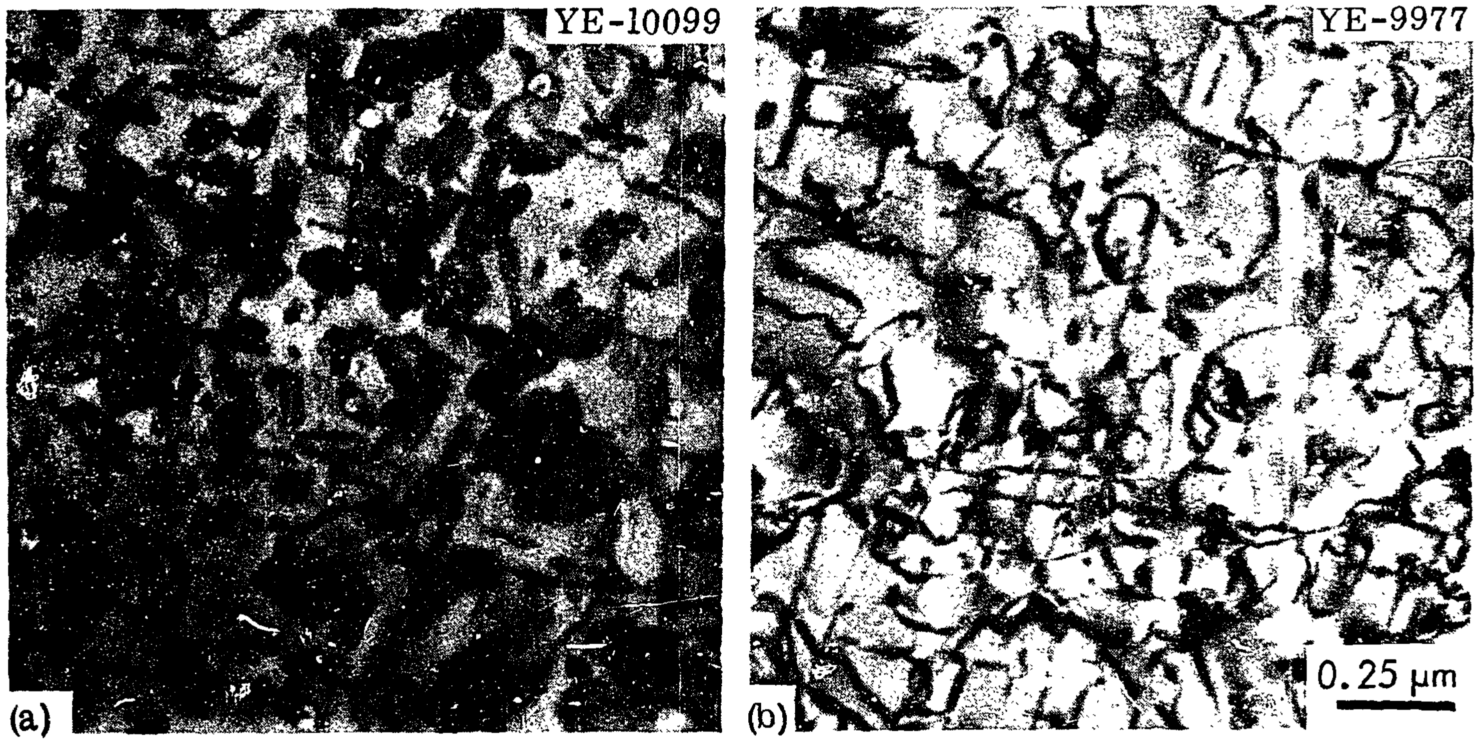

Fig. 2. Dislocation Structure Formed at Two Different Irradiation Temperatures. (a) Faulted loops formed at 535°C, 1.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV) and (b) unfaulted loops and dislocation network formed at 590°C, 3.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV).

of diffraction patterns from similar precipitates formed in type 316 stainless steel has shown them not to be one of the conventional precipitate structures found in stainless steels. A second type of precipitate having a platelike morphology was also present but in a much lower concentration. At the highest irradiation temperature examined in this study, about 820°C, small cavities were present on the grain boundaries as well as in the matrix. Because of their location on grain boundaries and relatively small size, we believe that these cavities are equilibrium helium bubbles resulting from (n,α) reactions. The bubbles within the grains, which averaged about 100 Å in diameter, were present in a concentration of about $10^{13}/\text{cm}^3$. Perhaps more important with regard to mechanical properties are the grain boundary bubbles, which are slightly larger and present in a concentration on the order of $10^{10}/\text{cm}^2$.

Below irradiation temperatures of about 550°C, the interstitials precipitated as Frank loops as illustrated in Fig. 2(a). Like the bubbles, the loops increased in size and decreased in concentration with increasing irradiation temperature. Above about 550°C, they unfaulted and apparently were able to glide and interact to form a loosely organized dislocation structure as shown in Fig. 2(b). At constant fluence the dislocation density decreased with increasing irradiation temperature.

Statistics describing the defect structures present in the specimens tested in this study are given in Table 1. A more complete description of radiation-induced defect configurations in type 304 stainless steel can be found in refs. 2–4.

Void formation is believed[5] to occur because of a delicate imbalance in the vacancy and interstitial concentrations caused by the preferential drift of interstitials to dislocations because of the stronger attraction of dislocations for interstitials than vacancies. Microstructural changes

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[3] E. E. Bloom, *An Investigation of Fast Neutron Radiation Damage in an Austenitic Stainless Steel*, ORNL-4580 (August 1970).

[4] E. E. Bloom, J. O. Stiegler, and C. J. McHargue, "Radiation Damage in Annealed Type 304 Stainless Steel," to be published.

[5] R. Bullough and R. C. Perrin, p. 317 in *Irradiation Effects on Structural Alloys for Nuclear Reactor Applications, Spec. Tech. Publ. 484*, American Society for Testing and Materials, Philadelphia, March 1971.

Table 1. Defect Statistics in Irradiated Stainless Steels

Preirradiation Condition	Irradiation Temperature (°C)	Neutron Fluence (> 0.1 MeV) (neutrons/cm^2)	Void Concentration (voids/cm^3)	Mean Void Diameter (Å)	Dislocation Concentration
		$\times 10^{22}$			
		Type 304 Stainless Steel			
1 hr at 1040°C	440	1.5	1.1×10^{15}	197	1×10^{16} est[a]
1 hr at 1040°C	450	2.5	2.0×10^{15}	193	
1 hr at 1040°C	535	1.5	2.3×10^{14}	240	2×10^{15}[a]
1 hr at 1040°C	590	2.5	3.7×10^{13}	175	
1 hr at 1040°C	590	3.5	5.5×10^{13}	353	2×10^{10}[b]
1 hr at 1040°C	820	2.5			
1 hr at 1040°C	*820*	*3.5*	$\sim 1 \times 10^{13}$[c]		
10% cold work	450	2.5	2.1×10^{14}	170	
		Type 304 + 0.15% Ti Stainless Steel			
1 hr at 1040°C	450	1.3	1.1×10^{15}	140	
1 hr at 925°C	590	2.0	5.8×10^{13}	342	
1 hr at 925°C	590	3.0	1.2×10^{14}	762	
1 hr at 925°C	820	3.0			

[a] Faulted dislocation loops, loops/cm^3.

[b] Dislocation network, cm/cm^3.

[c] Helium bubble concentration.

such as cold working that increase the density of sinks would be expected to alter defect configurations, concentrations, and sizes. There now is ample evidence that this is the case,[6–8] although not enough systematic information is available to describe the effects as completely as for annealed materials. At least at low fluences cold working inhibits the formation of voids. Stiegler and Bloom[7] found that in the type 304 stainless steel discussed previously the introduction of 10% cold work prior to irradiation substantially reduced swelling at an irradiation temperature of 450°C and a fluence of 2.5×10^{22} neutrons/cm^2. As can be seen from the microstructural data also presented in Table 1, the reduction in swelling was accomplished through an order of magnitude reduction in void concentration and a very slight reduction in mean void size. No faulted loops were evident in this specimen. Evidently the deformation-induced dislocation structure served as the primary interstitial sink. Additional effects have been observed at higher temperatures and fluences,[6,8] but in this report mechanical properties results will be limited to the single specimen described above.

It is also clear from examination of data in the literature[9,10] that composition is an important variable influencing the numbers and sizes of voids and loops. In this study, specimens of type 304 stainless steel modified by the addition of 0.15% Ti were annealed for 1 hr at 1040 or 925°C and then irradiated in EBR-II under conditions nearly

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[6] C. Cawthorne, E. J. Fulton, J. I. Bramman, G.A.B. Linekar, and R. M. Sharpe, "Electron Microscope Observations of Voids in Cladding Materials Irradiated in DFR," p. 35 in *Voids Formed by Irradiation of Reactor Materials*, (Proceedings of the British Nuclear Energy Society European Conference held at Reading University on March 24 and 25, 1971).

[7] J. O. Stiegler and E. E. Bloom, "The Effect of Thermo-Mechanical Treatments on Void Formation in Irradiated Stainless Steel," to be published in *Journal of Nuclear Materials*.

[8] J. L. Straalsund and H. R. Brager, paper presented at the International Conference on Radiation-Induced Voids in Metals, State University of New York, Albany, June 9–11, 1971 (to be published in the proceedings).

[9] T. Lauritzen, A. Withop, and U. E. Wolff, *Nucl. Engr. Design* 9: 265 (1969).

[10] E. E. Bloom and J. O. Stiegler, "A Comparison of Irradiation Induced Swelling and Void Formation in Two Austenitic Stainless Steels," *J. Nucl. Mater.* 35: 244–246 (May 1970).

identical to those described above for the standard type 304 stainless steel. The 925°C annealing treatment was emphasized in this study because in previous work[11,12] it was found to maximize the creep ductility after irradiation at temperatures above the range for displacement damage in which helium bubbles are thought to enhance grain-boundary fracture.

The 925°C preirradiation annealing treatment produced a relatively fine grain size (about 30 μm average diameter) and a high concentration of coherent precipitate particles as shown in Fig. 3. After irradiation at 590°C to a fluence of 2.0×10^{22} neutrons/cm^2, stringered arrays of noncoherent precipitates were found that apparently had descended from the original coherent precipitates. Most had voids attached as can be seen in Fig. 3(b). At the same irradiation temperature but at a higher fluence, 3×10^{22} neutrons/cm^2, considerable growth of the voids had occurred and all voids were attached to precipitate particles. In addition, as shown in Fig. 3(c), large voids were associated with large precipitate particles suggesting that at least some of the impurities involved in the precipitates were dragged with the vacancies to the voids.

Void statistics for the titanium-modified steel are also given in Table 1 where they can be compared with values for the standard alloy. At 440°C void sizes in the two are comparable but void concentrations and thus swelling are about a factor of 4 lower in the modified steel. At 590°C, however, both void concentrations and sizes are greater in the titanium-modified alloy. These observations may be interpreted in terms of a shift of the range of void formation to higher temperatures by the addition of 0.15% Ti to type 304 stainless steel.

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[11] W. R. Martin and J. R. Weir, "Solutions to the Problems of High-Temperature Irradiation Embrittlement," pp. 440–457 in *Effects of Radiation on Structural Metals, Spec. Tech. Publ. 426*, American Society for Testing and Materials, Philadelphia, 1967.

[12] E. E. Bloom and J. R. Weir, "Development of Austenitic Stainless Steels with Improved Resistance to Elevated-Temperature Irradiation Embrittlement," pp. 261–289 in *Irradiation Effects in Structural Alloys for Thermal and Fast Reactors, Spec. Tech. Publ. 457*, American Society for Testing and Materials, Philadelphia, 1969.

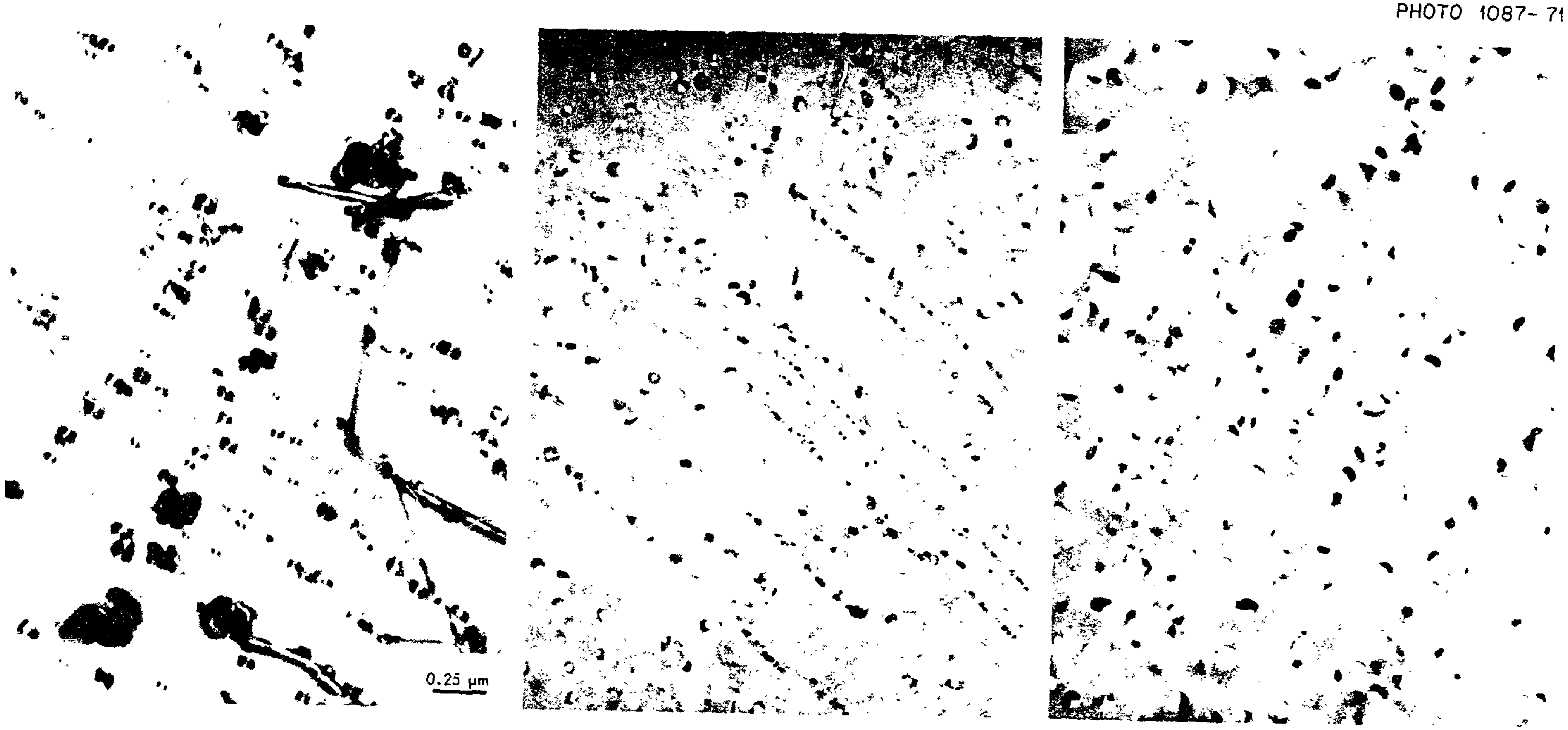

Fig. 3. Association of Voids and Precipitates in Type 304 + 0.2% Ti Stainless Steel. (a) Annealed 1 hr at 925°C, (b) irradiated at 590°C to 2.0 × 10^{22} neutrons/cm^2 (> 0.1 MeV), and (c) irradiated at 590°C to 3.0 × 10^{22} neutrons/cm^2 (> 0.1 MeV).

Tensile Properties

The effects of irradiation on the tensile properties of annealed type 304 stainless steel are shown in Fig. 4. Specimens irradiated and tested at 450°C exhibited a yield strength which was approximately a factor of 5 higher than the value for unirradiated specimens. With increasing temperature the yield strength of the irradiated material decreased and approached the value for unirradiated specimens at 700 to 750°C. Specimens irradiated at 590°C and tested at 450, 550, and 650°C all exhibited similar yield strengths. The ultimate tensile strengths were similar for irradiated and unirradiated specimens except at the lowest test temperature (450°C) where the irradiated values were somewhat higher. Ductility as measured by uniform strain, total elongation,

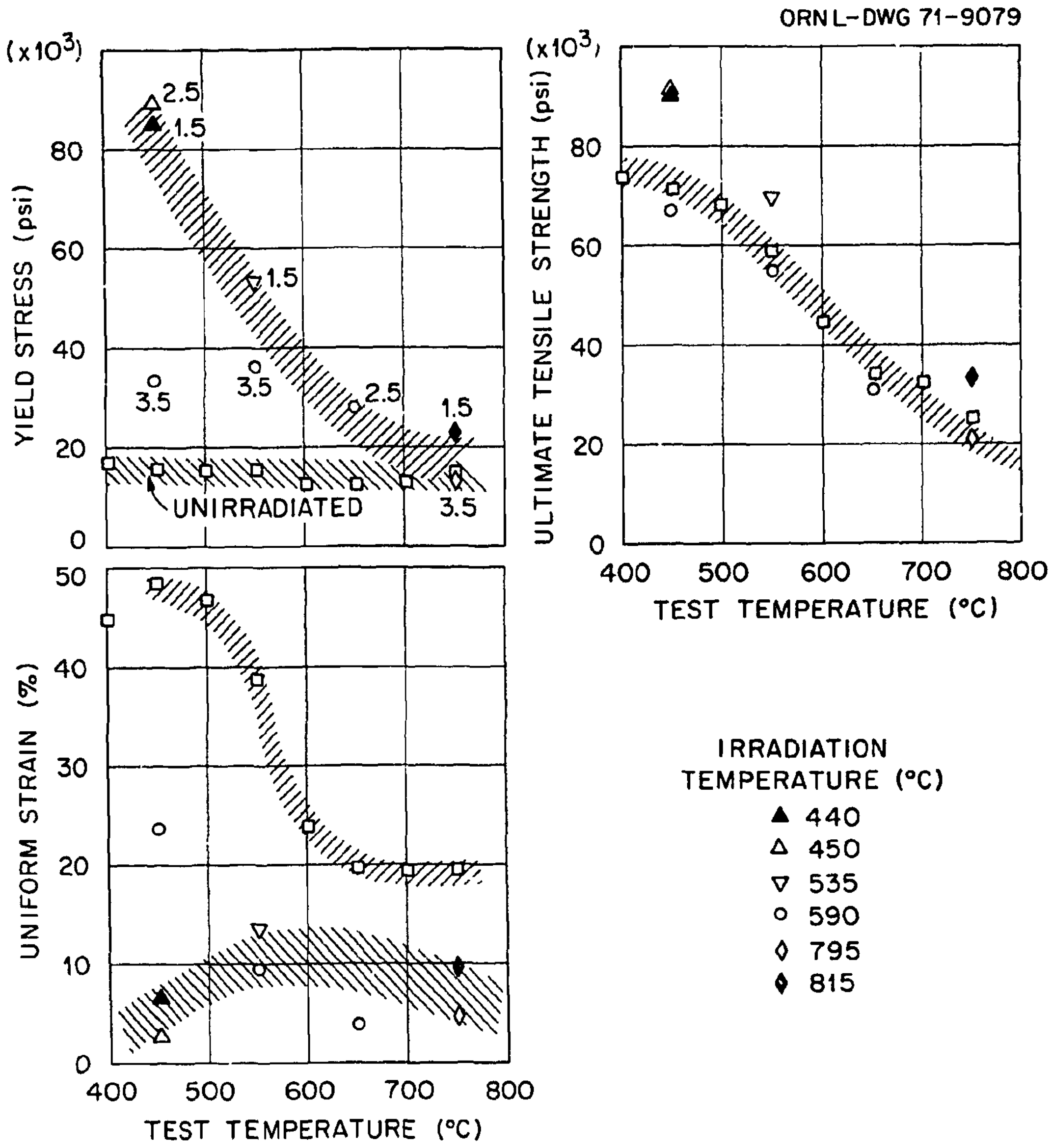

Fig. 4. Effect of Fast Neutron Irradiation on the Tensile Properties of Annealed Type 304 Stainless Steel. The numbers by each data point are fluences × 10^{-22} (> 0.1 MeV).

and reduction in area was reduced at all temperatures. The reduction in ductility became more severe with increasing fast neutron fluence. Note, for example, that in Fig. 4 the uniform strain for specimens irradiated to 1.5 and 3.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV) lie at the upper and lower edges of the scatter band, respectively. At 450°C the reduced uniform strain resulted from a reduced work hardening coefficient and thus plastic instability occurred early in the deformation. Metallographic examination indicated that the fractures were transgranular with a large amount of deformation having occurred in the immediate vicinity of the failure. At 750°C the stress-strain characteristics were unaffected by irradiation. At these higher temperatures the fractures were predominantly intergranular with uniform strain, total elongation, and reduction in area well below the unirradiated values. In specimens which were irradiated and tested at 450°C, the deformation was confined to planar bands within the matrix as shown in Fig. 5(a). A large amount of deformation had occurred within these bands. In some instances shear strains of 2 to 3 were measured from observations of displacement of intersecting bands. It did not appear that the irradiation-produced defects were eliminated by the deformation as has been observed in some metals,[13] Voids which were

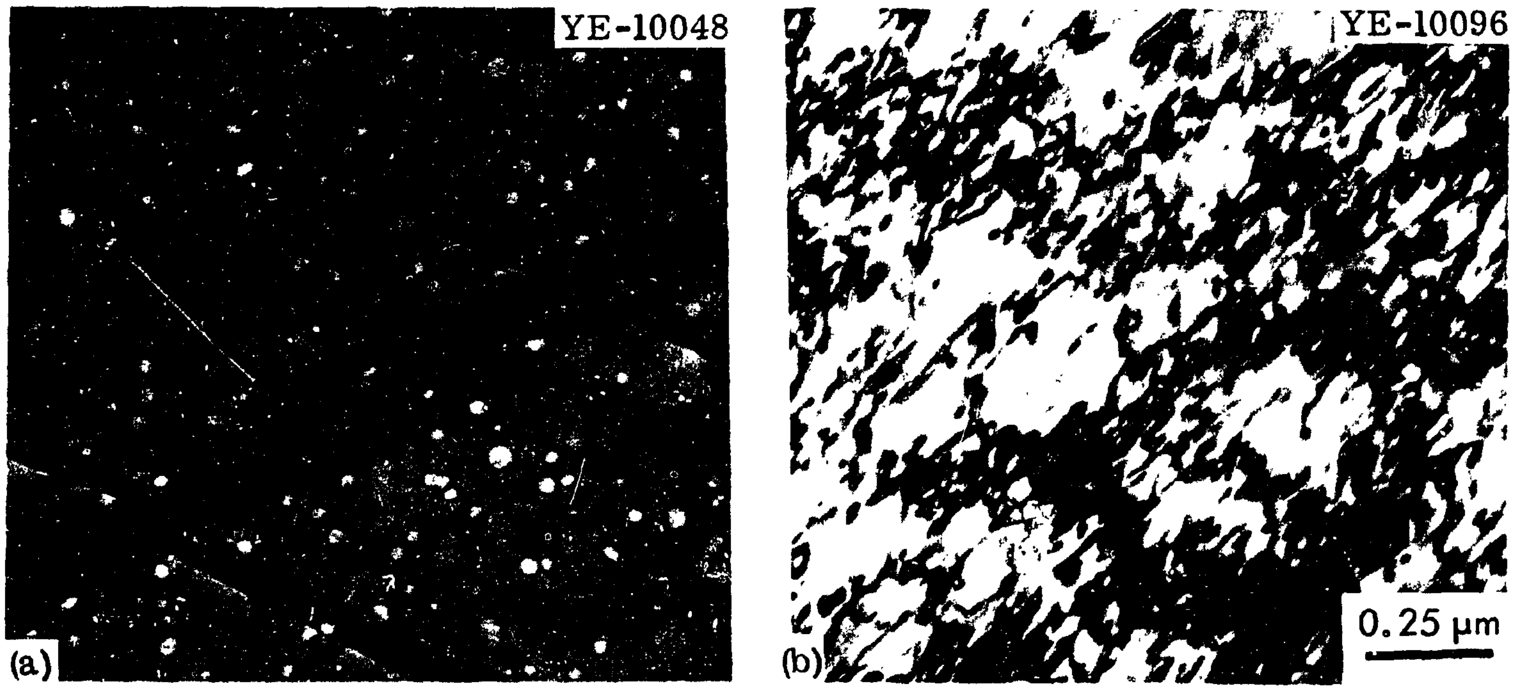

Fig. 5. Microstructures Illustrating the Different Deformation Structures in Specimens. (a) Irradiated and tested at 450°C and (b) irradiated at 590°C and tested at 650°C.

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[13] I. G. Greenfield and H.G.F. Wilsdorf, *J. Appl. Phys.* 32: 827 (1967).

located within the bands were distorted in shape or sheared by the large amount of strain. The dislocation structure in the bands after deformation was very complex and could not be analyzed in detail. No evidence was found to indicate that deformation had occurred between the bands. In a specimen irradiated at 590°C and tested at 650°C the deformation was similar to that of an unirradiated specimen with the formation of dislocation tangles and cells as shown in Fig. 5(b).

Postirradiation tensile properties for annealed and 10% cold-worked type 304 stainless steel irradiated and tested at 450°C are given in Table 2. As discussed above, irradiation of annealed type 304 stainless steel caused a large increase in yield strength, a small increase in ultimate tensile strength, and large reductions in uniform strain and total elongation. Cold working 10% caused similar changes in these properties and irradiation of the cold-worked material produced only minor changes. A slightly different trend was noted for the reduction in area and fracture stress. In the unirradiated specimens the reduction in area and fracture stress were about the same for the annealed

Table 2. Tensile Properties of Annealed and 10% Cold-Worked
Type 304 Stainless Steel Irradiated and Tested at 450°C

Condition	Stress, psi		Elongation, %		Reduction in Area (%)	Fracture Stress (psi)
	Yield	Ultimate	Uniform	Total		
Annealed unirradiated	15,500	71,700	47.3	51.8	60.7	130,300
Annealed irradiated[a]	85,300	90,500	6.5	11.3	35.9	102,000
10% cold work unirradiated	89,500	96,500	4.5	9.4	53.3	146,400
10% cold work irradiated[b]	89,500	97,000	5.0	6.9	19.6	106,500

[a] 1.5×10^{22} neutrons/cm^2 (> 0.1 MeV).

[b] 2.5×10^{22} neutrons/cm^2 (> 0.1 MeV).

and 10% cold-worked samples. Irradiation caused a reduction in both of these parameters.

The addition of 0.15% Ti to type 304 stainless steel did not significantly alter the postirradiation tensile strength properties as can be seen by comparing values given in Table 3 with corresponding values for the standard material plotted in Fig. 4. However, ductility, as measured by uniform strain, total elongation, or reduction in area, was somewhat higher for the titanium-modified specimens irradiated and tested at temperatures above about 450°C.

Table 3. Postirradiation Tensile Properties of Titanium-Modified Type 304 Stainless Steel

Fast Neutron Fluence (> 0.1 MeV) (neutrons/cm^2)	Temperature, °C		Stress, psi		Elongation, %		Reduction in Area (%)
	Irradiation	Test	Yield	Ultimate	Uniform	Total	
$\times 10^{22}$							
1.3[a]	450	450	68.0	77.0	6.6	11.9	28.6
2.0[b]	590	550	39.6	53.9	19.1	24.6	37.7
2.0[b]	590	650	35.8	40.7	8.8	30.4	34.9
3.0[b]	820	750	15.5	17.4	10.0	44.2	47.0

[a] Annealed 1 hr at 1040°C, tested at 0.01/min.

[b] Annealed 1 hr at 925°C, tested at 0.002/min.

Creep-Rupture Properties

The effects of irradiation to fluences in the range 1.5×10^{22} neutrons/cm^2 (> 0.1 MeV) on the creep-rupture properties of annealed type 304 stainless steel are shown in Fig. 6. At 550 and 600°C no major change in the rupture life was found, but at these two temperatures the creep rates of the irradiated samples were lower, by approximately a factor of 2, than those exhibited by the unirradiated specimens. Note, however, that the unirradiated material was tested at stresses above the yield stress and thus strain occurred during application of the load.

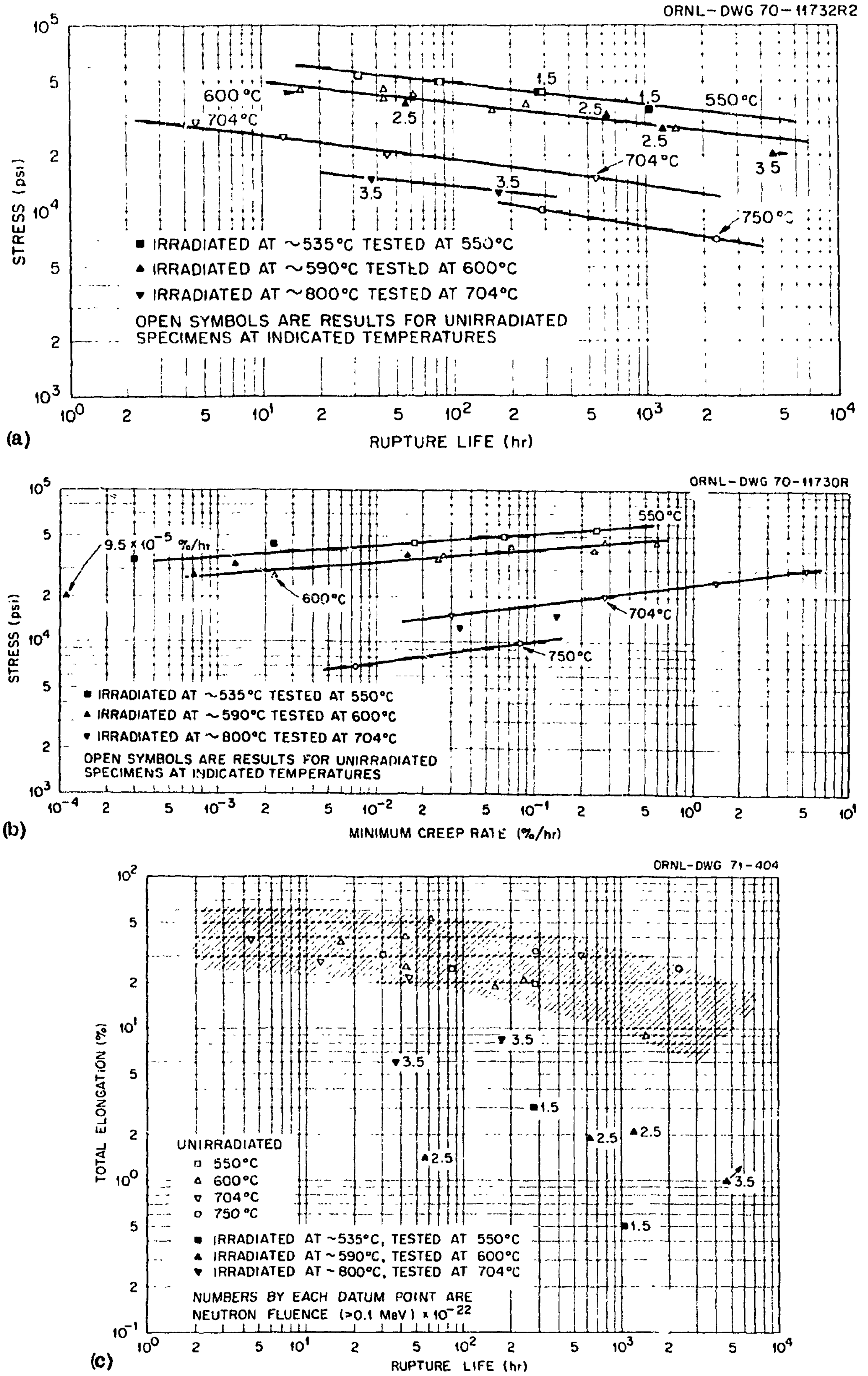

Fig. 6. Effect of Fast Neutron Irradiation on the Creep-Rupture Properties of Annealed Type 304 Stainless Steel. (a) Rupture life, (b) creep rate, and (c) total elongation.

For example, at 550°C it is estimated that 13% strain occurred upon application of 55,000 psi stress. In comparison, the yield stress of the irradiated specimens at 550°C was about 54,000 psi and thus essentially no plastic strain occurred on loading. In essence, the creep rate results indicate that the void and dislocation loop structures shown in Figs. 1 and 2 decreased the creep rate by about the same amount as the loading deformation which was introduced during application of the stress to the unirradiated specimens. The creep curve and results of transmission and scanning electron microscopy examinations of a specimen which was irradiated at 535°C to 1.5×10^{22} neutrons/cm^2 and tested at 550°C and 35,000 psi are summarized in Fig. 7. Before test, the specimen contained 2.3×10^{14} voids/cm^3 with a mean void diameter of 230 Å and about 2×10^{15} faulted dislocation loops/cm^3. The unstressed shoulder portion of the tested specimen contained about 9.4×10^{13} voids/cm^3 with a mean diameter of 190 Å and a dislocation structure consisting of faulted loops, perfect loops (presumably produced by thermally activated unfaulting), and segments of dislocation lines which could have resulted from the climb and interaction of perfect loops. In the stressed gage section the void concentration was 5×10^{13} voids/cm^3, and the mean void diameter was 200 Å. No faulted loops were present in this portion of the specimen, and the dislocation density was slightly higher than in the unstressed shoulder. The faulted loops could have been removed by interaction with glide dislocations as shown by Strudel and Washburn.[14] These results indicate that the irradiation-produced microstructure is quite stable to annealing, whether stressed or unstressed, when held at or near the irradiation temperature.

Specimens which were irradiated and tested at 550 and 600°C exhibited total elongations in the range 0.5 to 3.0% [Fig. 6(c)]. For these irradiation and test conditions the fractures were completely intergranular (Fig. 7). Optical metallography of tested specimens showed few if any secondary grain-boundary cracks or incipient fractures, thus indicating that a crack, once initiated, propagates rapidly to cause failure.

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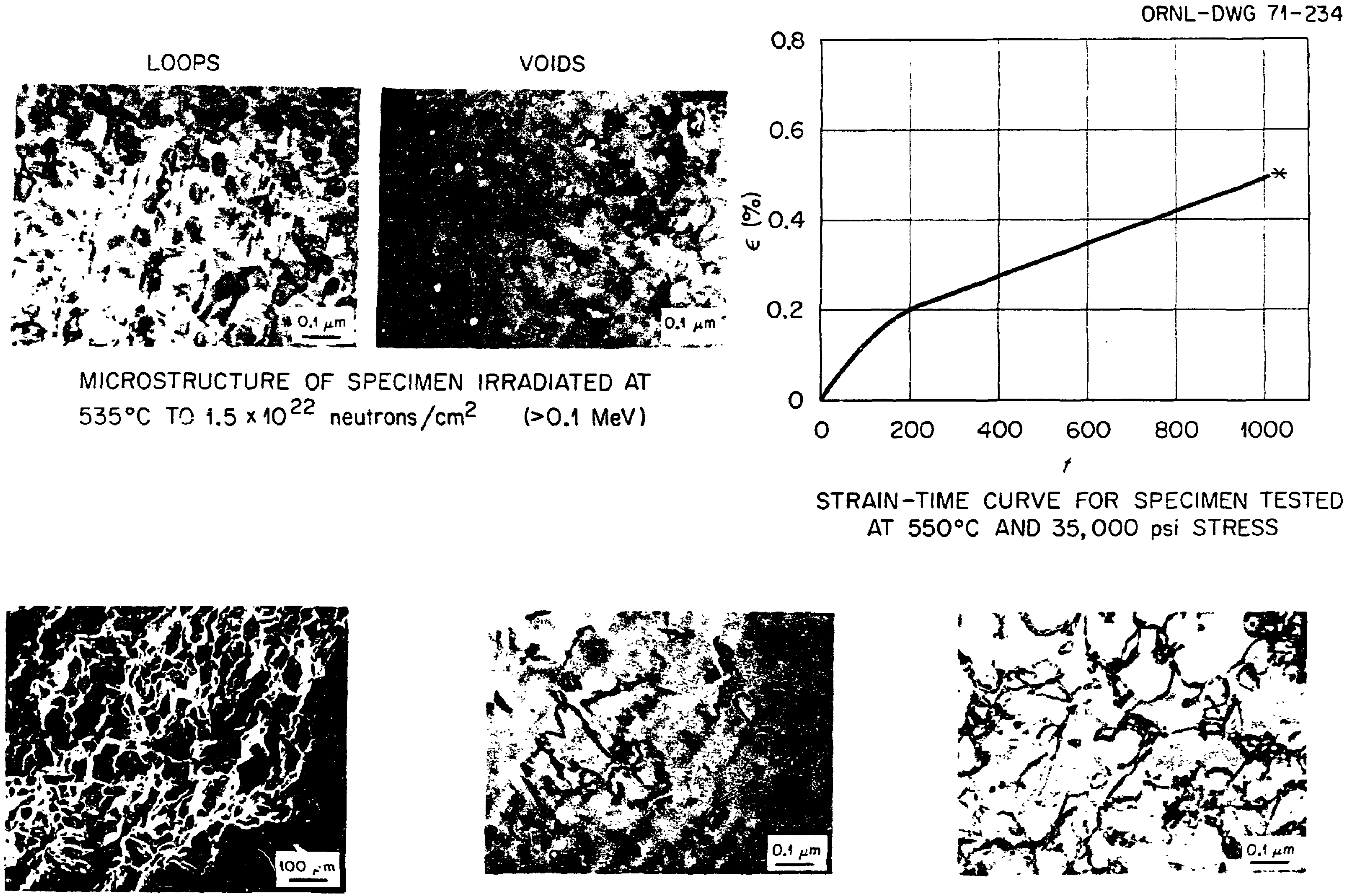
[14] J. L. Strudel and J. Washburn, *Phil. Mag.* 9: 491 (1968).



MICROSTRUCTURE OF SPECIMEN IRRADIATED AT 535°C TO 1.5×10^{22} neutrons/cm^2 (>0.1 MeV)

STRAIN-TIME CURVE FOR SPECIMEN TESTED AT 550°C AND 35,000 psi STRESS

INTERGRANULAR FRACTURE OF TESTED SPECIMEN

MICROSTRUCTURE IN UNSTRESSED SHOULDER REGION

MICROSTRUCTURE IN STRESSED GAUGE SECTION

Fig. 7. Creep Rupture of Type 304 Stainless Steel after Irradiation in EBR-II.

Specimens which were irradiated at 800°C and tested at 704°C exhibited ductilities which were lower than those for the unirradiated samples but higher than those for specimens irradiated and tested at 550 to 600°C. At 704°C the creep rate was higher for the irradiated specimens. The combination of increased creep rate and reduced ductility led to reductions in rupture life of about an order of magnitude.

The results of creep-rupture tests for the titanium-modified type 304 stainless steel after irradiation are listed in Table 4. At 600°C, the rupture life of the modified alloy was about one-fourth that of the standard material irradiated in the same experiment at the same temperatures; its minimum creep rate was about ten times higher. These differences in strength properties were a result of the finer grain size in the titanium-modified type 304 stainless steel (annealed 1 hr at 925°C before irradiation) as compared to that in the standard type 304 stainless steel (annealed 1 hr at 1050°C before irradiation). The ductility of the modified alloy after irradiation, in terms of elongation at the end of second-stage creep, total elongation, or reduction in area, was significantly higher than that of the standard type 304 stainless steel. Results of metallographic examination of a specimen of the modified alloy irradiated at about 590°C to 3.0×10^{22} neutrons/cm^2 and then creep tested at 600°C and 27,500 psi stress are summarized in Fig. 8. Note from the curve of strain versus time that the material underwent about 11% strain before going into third-stage creep as compared to zero third-stage creep and 0.5% strain to failure in the standard alloy (see Fig. 7). Transmission electron microscopy of specimens from the stressed gage section revealed that a dislocation cell structure had formed with the voids and precipitate particles acting as pinning points. Most significant was the observation by means of scanning electron microscopy that the specimen had failed in a very ductile, predominantly transgranular mode. This can be compared to the intergranular fracture of standard type 304 stainless steel which is shown in Fig. 7.

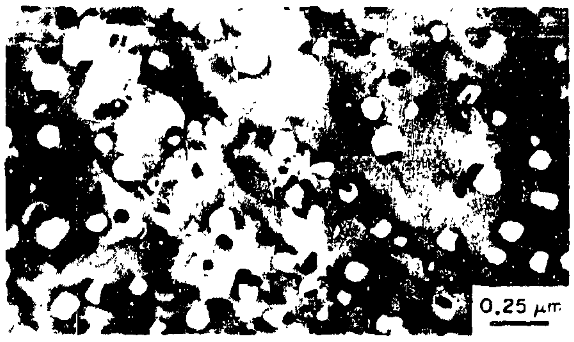
DISCUSSION

The void, dislocation, and precipitate structures which were formed in annealed type 304 stainless steel during irradiation to neutron fluences

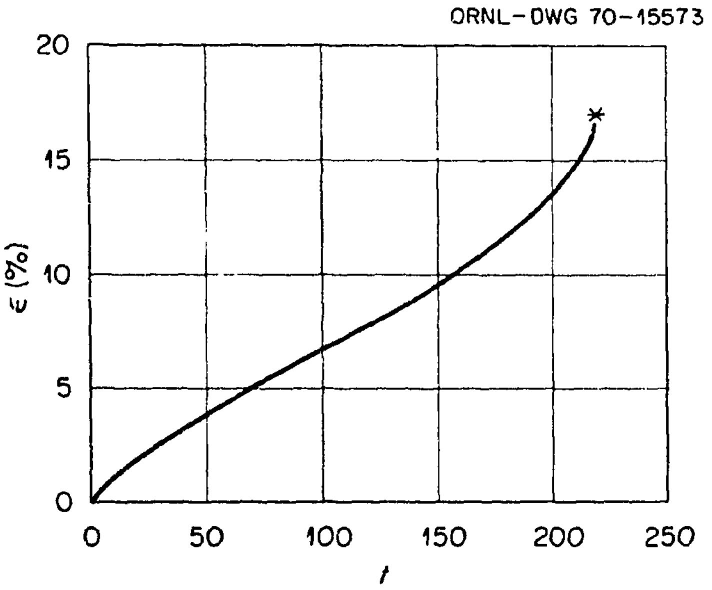
Table 4. Creep-Rupture Properties of Titanium-Modified Type 304 Stainless Steel After Irradiation[a]

Fast Neutron Fluence (> 0.1 MeV) (neutrons/cm^2)	Temperature, °C		Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation, %		Reduction in Area (%)
	Irradiation	Test				At End of Second Stage	Total	
$\times 10^{22}$								
2.0	590	600	32,500	146.1	0.072	5	21.1	23
3.0	590	600	27,500	220.1	0.054	11	17.0	22
3.0	590	600	20,000	3046.7	0.0019	9	9.9	16
3.0	800	704	15,000	49.9	0.49	20	35.1	24.5
3.0	800	704	12,500	287.4	0.076	13	29.8	30.1

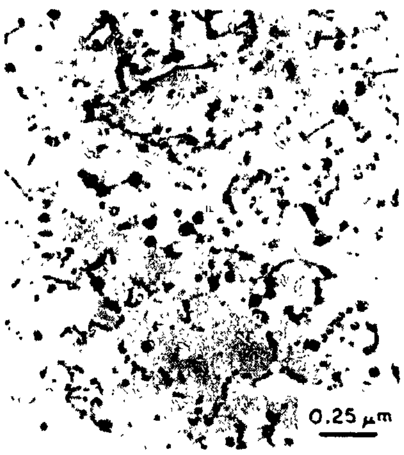
[a] Specimens were annealed 1 hr at 925°C before irradiation in the EBR-II.



MICROSTRUCTURE OF M304 STAINLESS STEEL IRRADIATED AT 590°C TO 3.0×10^{22} neutrons/cm^2 (>0.1 MeV)

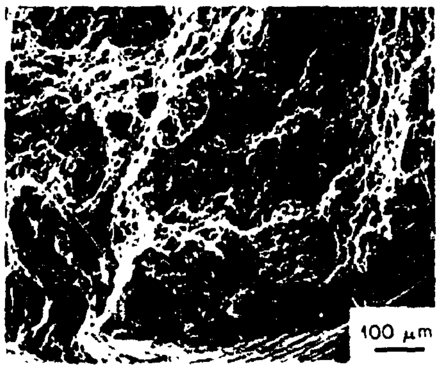


STRAIN-TIME CURVE FOR SPECIMEN TESTED AT 600°C AND 27,500 psi STRESS



DISLOCATION STRUCTURE IN UNSTRESSED SHOULDER OF TESTED SPECIMEN

DISLOCATION STRUCTURE IN GAUGE SECTION OF TESTED SPECIMEN



FRACTURE SURFACE

Fig. 8. Creep-Rupture Properties of Type 304 + 0.15% Ti Stainless Steel after Irradiation in the EBR-II.

of 1.5 to 3.5 × 10^{22} neutrons/cm^2 (> 0.1 MeV) at temperatures between 400 and 820°C have been characterized by transmission electron microscopy. Changes in tensile properties were in qualitative agreement with these microstructural observations. The large increase in yield stress at the lower irradiation and test temperature was a result of the complex defect structures formed during irradiation. The increase in yield stress became less at higher irradiation temperatures due to the decreasing defect concentrations. The contributions of the various defects in producing the observed increase in yield stress are difficult to assess. Using the calculations of Foreman[15] and Fleischer,[16] the increase in yield stress ($\Delta\sigma_\ell$) due to loops is approximately

$$\Delta\sigma_\ell = \frac{Gb(N_\ell d_\ell)^{1/2}}{3.7} , \qquad (1)$$

where N_ℓ is the loop density, d_ℓ is the loop diameter, G is the shear modulus, and b is the Burgers vector. For the specimen irradiated at 440°C and tested at 450°C, the predicted increase in yield stress due to loops is approximately 21,000 psi. Using the results of Coulomb[17] the hardening due to cavities is given by $\Delta\sigma_v = 2Gb/\ell$, where $\ell = 1/(N_v d_v)^{1/2}$ is the void spacing (d_v is the mean void diameter and N_v is the void concentration). For the above specimen, $\Delta\sigma_v$ due to voids would be about 9000 psi. Using the expression[18]

$$\Delta\sigma_{total} = (\Delta\sigma_\ell^{\,2} + \Delta\sigma_v^{\,2})^{1/2} , \qquad (2)$$

the predicted yield stress increase is 24,000 psi which is about a factor of 2 smaller than observed. In specimens irradiated and tested at higher temperatures, the observed increase in yield stress was in close agreement with the value calculated from dislocation network and precipitate

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[15] A.J.E. Foreman, *Phil. Mag.* 17: 353 (1968).

[16] R. L. Fleischer, *Acta Met.* 10: 835 (1962).

[17] P. Coulomb, *Acta Met.* 7: 556 (1959).

[18] T. J. Koppenaal and D. Kuhlmann-Wilsdorf, *Appl. Phys. Letters* 4: 59 (1964).

hardening. The poorer agreement at 450°C may be due to small defects or precipitate particles which contribute to the hardening but are invisible by transmission electron microscopy due to the complex dislocation structure. Additionally, Eq. (1) was not derived for the present case. Its use has been sanctioned by reasonable agreement with experiment in several previous cases.[18—20] The tensile properties of specimens which were cold worked 10% prior to irradiation at 450°C were similar to those of specimens which were irradiated in the annealed condition. This is the expected result because the dislocation structure controls the strength whether the dislocations are introduced by irradiation or cold work. At higher irradiation temperatures (550 to 750°C), where recovery or recrystallization may occur, the behavior of annealed and cold-worked material is expected to be considerably different.

Holmes et al.[19,21] have shown that in annealed type 304 stainless steel irradiated at about 540°C to 1.4×10^{22} neutrons/cm^2 (> 0.18 MeV) and tested in the range 20 to 871°C full recovery of the yield stress does not occur until a test temperature of 816°C is reached. In the present study no significant yield stress increase was found above 700 to 750°C. The importance of testing at or near the irradiation temperature when investigating the effects of irradiation on mechanical properties is thus apparent.

The most significant effect of irradiation on mechanical properties is the loss of ductility. For irradiation temperatures below 500°C, a large reduction in uniform and total tensile elongation occurs. This effect can be related to the reduced work-hardening coefficient since necking or plastic instability occurs when the true strain equals the work-hardening coefficient. The flow stress is initially high due to the presence of the irradiation-produced defects. Once it is initiated, slip remains confined to narrow bands. The exact mechanism for the

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[19] J. J. Holmes, R. E. Robbins, J. L. Brimhall, and B. Mastel, *Acta Met.* 16: 955 (1968).

[20] P. J. Barton and P.R.B. Higgins, p. 362 in *Irradiation Effects in Structural Alloys for Nuclear Reactor Applications, Spec. Tech. Publ. 484*, American Society for Testing and Materials, Philadelphia, March 1971.

[21] J. J. Holmes, R. E. Robbins, and J. L. Brimhall, *J. Nucl. Mater.* 32: 330 (1969).

formation of these bands is not understood. Similar features are observed in unirradiated stainless steels when the material has been strained in excess of about 10% at room temperature. Other investigators have reported that these bands are clusters of stacking faults produced by slip on closely spaced slip planes,[22] that they are mechanical twins,[23] or that they are a hexagonal epsilon phase.[24] Complications in the diffraction patterns from the faulted loops prevented a positive identification in this case. The bands normally are not found in materials deformed at elevated temperatures. Evidently the strengthening from the radiation-induced defects promoted their occurrence. The bands observed in the present study were not channels in the sense that they had been swept free of dislocations and voids. It is possible that the slip dislocations weaken the irradiation-produced defects within these bands, and subsequent deformation does not produce sufficient work hardening to cause the flow stress within the bands to reach that of the matrix.

Below about 500°C, the material fractures in a ductile transgranular mode in both the irradiated and unirradiated conditions. Lower reductions in area and fracture stresses were, however, observed in the irradiated specimens. At low temperatures (e.g., 450°C) the fractures may well be initiated in a manner similar to that proposed by Rogers[25] and Puttick.[26] Cavities initially form in regions of high stresses created by inhomogeneous deformation or at nonmetallic inclusions due to inhomogeneous strain at the interface. As straining proceeds the cavities grow, eventually linking to form a central crack. This crack propagates as deformation is concentrated at its tip with large numbers of cavities being nucleated in the region of heavy shear strain. In irradiated material, cracks may have initiated and propagated by the same process, but at lower overall strains due to the high concentration of strain in the observed channels.

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[22] B. Weiss and R. Stickler, Westinghouse Research Laboratories Scientific Paper 70-1D4-STABL-P1, July 1970.

[23] J. A. Maza, *J. Iron Steel Inst. (London)* 204: 783 (1966).

[24] P. A. Blenkinsop and J. Nutting, *J. Iron Steel Inst. (London)* 205: 953 (1967).

[25] H. C. Rogers, *Trans. Met. Soc. AIME* 218: 498 (1960).

[26] K. E. Puttick, *Phil. Mag.* 4: 964 (1960).

In the range 500 to 600°C the tensile properties are affected less than at lower temperatures because of the reduced concentrations of voids and dislocations. At these temperatures, however, very large reductions in creep-rupture ductility occur. It is within this temperature range that the effects of transmutation-produced helium are first observed.[27] The loss of ductility cannot, however, be attributed solely to effects of helium. First, specimens containing the same amount of helium exhibited higher ductility when irradiated and tested at higher temperatures (750°C) where void formation did not occur and the effects of helium are expected to be more pronounced. Secondly, as discussed previously,[28] specimens containing uniform concentrations of injected helium do not exhibit such large reductions in ductility as those found in the irradiated specimens. Scanning electron microscopy and optical metallography indicated that the fractures in the standard alloy were intergranular and that a crack, once initiated, propagated rapidly to cause failure. In irradiated specimens the regions adjacent to the grain boundaries were denuded of the damage structure. Deformation along the boundaries probably occurs in a similar fashion in unirradiated and irradiated specimens. When this deformation occurs, stresses are concentrated at constraints such as grain-boundary junctions. In unirradiated specimens, these stresses can be reduced by deformation within the matrix. For irradiated specimens, deformation in the matrix is impeded by the defect structure, and cracks are thus nucleated in high-stress regions. The propagation of these cracks along grain boundaries is likely to be enhanced by the presence of helium. At the highest irradiation temperatures the observations are consistent with the helium embrittlement phenomena.

The titanium-modified type 304 stainless steel is significantly more ductile at temperatures where helium embrittlement phenomena

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[27] D. R. Harries, *J. Brit. Nucl. Energy Soc.* 5: 74 (1966).

[28] E. 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.

predominate and also exhibits improved ductility under irradiation and test conditions where both displacement damage (i.e., voids and dislocations) and helium affect properties. The increased postirradiation ductility is a result of the decreased tendency for grain-boundary fracture.