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EFFECT OF FAST NEUTRON IRRADIATION ON THE CREEP-RUPTURE PROPERTIES OF TYPE 304 STAINLESS STEEL AT 600°C

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ABSTRACT

The creep-rupture properties of type 304 stainless steel have been determined at 600°C after irradiation at temperatures in the range 370 to 600°C to fast-neutron fluences of 1×10^{21} to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev). The microstructures were characterized by electron microscopy. Irradiation at 370 to 470°C caused a decrease in ductility and rupture life which became larger with increasing fast neutron fluence. The irradiated specimens fractured along the grain boundaries with no evidence of deformation within the matrix. A specimen irradiated at 410°C to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev) ruptured in 0.5 hr with 0.1% elongation as compared to the unirradiated rupture life of 185 hr and elongation of 19.7%. Specimens irradiated at 600°C to fluences from 2.5 to 3.5×10^{22} neutrons/cm^2 (> 0.1 Mev) exhibited decreased ductilities and creep rates and essentially the same rupture lives as unirradiated specimens.

The increased tendency for grain-boundary fracture, and thus reduced ductility and rupture life, appears to result from the effects of the irradiation-produced voids and dislocations upon the deformation processes which would be operative in an unirradiated specimen. The voids and dislocations prevent dislocation motion within the matrix. Regions along grain boundaries are denuded of these defects and grain-boundary sliding occurs, leading to stress concentrations and the initiation of grain-boundary cracks. The presence of helium at the boundaries may increase the rate of crack propagation.

INTRODUCTION

Austenitic stainless steels have several properties which make them attractive for use as fuel cladding and structural components in liquid metal fast breeder reactors (LMFBR). These alloys possess adequate high-temperature strength, are resistant to corrosion by liquid

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sodium (the reactor coolant), and are compatible with uranium-plutonium oxides (the proposed reactor fuel) at the projected operating temperatures of 350 to 650°C. Changes in mechanical and physical properties do occur, however, as a result of neutron irradiation. It is important that these changes do not endanger the safe or economical operation of the reactor system.

Changes in properties are a sensitive function of the irradiation temperature, the neutron fluence, and the energy spectrum, as well as the postirradiation test variables. It now appears that the irradiation damage as characterized by electron microscopy can be classified into three forms, depending on the irradiation temperature.[1] At low temperatures (< 0.37 T_m, 350°C, where T_m is the absolute melting temperature), the visible damage has the form of small defect clusters a few tens of angstroms in diameter. Such damage is observed at fast-neutron fluences as low as 1×10^{20} neutrons/cm^2 and does not change in form at least for fluences up to 3×10^{22} neutrons/cm^2 (refs. 2, 3). At intermediate temperatures (0.37 to 0.55 T_m, i.e., 350 to 650°C) the damage structure consists of voids (ranging up to a few hundred angstroms in diameter) and interstitial dislocation loops. The changes in this structure with

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[1]J. R. Weir, J. O. Stiegler, and E. E. Bloom, "Irradiation Behavior of Cladding and Structural Materials," pp. 189–224 in Proceedings of the National Topical Meeting on Fast Reactor Systems, Materials and Components, Cincinnati, Ohio, April 2–4, 1968, CONF-680419.

[2]C. Cawthorne and E. J. Fulton, "The Influence of Irradiation Temperature on the Defect Structures in Stainless Steel," p. 446 in The Nature of Small Defect Clusters, Vol. 2, ed. by M. J. Makin, Atomic Energy Research Establishment, Harwell, Report AERE-R5269, Her Majesty's Stationery Office, London, 1966.

[3]C. Cawthorne and E. J. Fulton, "Voids in Irradiated Stainless Steel," Nature 216, 515 (1967).

increasing fast-neutron fluence and irradiation temperature have been discussed in detail elsewhere.[2–7] At high temperatures, above approximately 650°C, the primary form of damage is helium bubbles formed by precipitation of helium produced by (n,α) transmutations. For LMFBR applications, irradiation damage at temperatures above approximately 350°C is of concern. For specimens irradiated and tested at intermediate temperatures, where the damage structure consists of voids and dislocation loops, the tensile yield strength is increased and the uniform elongation is reduced.[8–10] Holmes *et al.*[8,9] have investigated

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[4] S. D. Harkness and Che-Yu Li, "A Model for Void Formation in Metals Irradiated in a Fast-Neutron Environment," p. 189 in *Radiation Damage in Reactor Materials Vol. 2*, International Atomic Energy Agency, Vienna, 1969.

[5] T. T. Claudson, J. J. Holmes, J. L. Straalsund, and H. R. Brager, "Fast-Reactor Radiation Induced Changes in Cladding and Structural Materials," p. 165 in *Radiation Damage in Reactor Materials Vol. 2*, International Atomic Energy Agency, Vienna, 1969.

[6] J. O. Stiegler and E. E. Bloom, "The Effects of Large Fast-Neutron Fluences on the Structure of Stainless Steel," *J. Nucl. Mater.* **33**, 173 (1969).

[7] E. E. Bloom, *An Investigation of Fast Neutron Radiation Damage in an Austenitic Stainless Steel*, ORNL-4580 (August 1970), Ph.D. Thesis, The University of Tennessee.

[8] J. J. Holmes, R. E. Robbins, J. L. Brimhall, and B. Mastel, "Elevated Temperature Irradiation Hardening in Austenitic Stainless Steel," *Acta Met.* **16**, 955 (1968).

[9] J. J. Holmes, R. E. Robbins, and J. L. Brimhall, "Effect of Fast Reactor Irradiation on the Tensile Properties of 304 Stainless Steel," *J. Nucl. Mater.* **32**, 330 (1969).

[10] R. Carlander, S. D. Harkness, and F. L. Yaggee, "Fast Neutron Effects on Type 304 Stainless Steel," *Nucl. Appl.* **7**, 67 (1969).

the postirradiation tensile properties of type 304 stainless steel irradiated at 540 ± 50°C to 1.1×10^{22} neutrons/cm^2 (> 0.1 Mev). At test temperatures below 0.5 T_m, the ductility loss was caused by the onset of the plastic instability (local necking) induced by the increased flow stress and reduced work-hardening rates. Above 0.5 T_m, helium embrittlement was thought to control ductility. Carlander et. al.[10] have investigated the change in tensile properties as a function of fast-neutron fluence and postirradiation test temperature. For specimens irradiated at temperatures in the range 370 to 470°C and tensile tested at 450°C the changes in yield strength and uniform elongation were found to saturate at about 2×10^{22} neutrons/cm^2 (> 0.1 Mev).

Previous investigations in which specimens were irradiated in thermal reactors have shown that at test temperatures above about 550°C the transmutation-produced helium caused a significant reduction in the ductility, particularly at the low strain rates encountered in creep-rupture tests.[11,12] This loss of ductility results from the increased rate of grain-boundary crack initiation and propagation due to helium at the grain boundaries. The effect of irradiation to high fast-neutron fluences on the creep-rupture properties has not been investigated. Of particular interest are the changes in properties which occur when the specimen contains the void and dislocation loop structure formed at irradiation temperatures below about 650°C and when the test is conducted at a temperature where helium embrittlement is an important factor (i.e., above about 550°C). The objective of this work was to study the postirradiation creep-rupture properties in specimens which were irradiated under different conditions in order to relate the changes in creep-rupture properties to the irradiation-produced microstructure.

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[11] D. R. Harries, "Neutron Irradiation Embrittlement of Austenitic Stainless Steels and Nickel Base Alloys," J. Brit. Nucl. Energy Soc. 5, 74 (1966).

[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.

EXPERIMENTAL PROCEDURE

Sheet specimens of type 304 stainless steel, having the dimensions shown in Fig. 1, were machined from an irradiated Experimental Breeder Reactor-II (EBR-II) safety-rod thimble. The thimble, which was a hexagonal tube measuring about 3.2 cm across each flat, 0.10 cm thick, and 116.6 cm long, was placed in the reactor in the annealed condition and had an average grain diameter of 0.045 mm. It was irradiated in a row 3 position of the reactor to a peak fluence of 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev). There was a gradient in both neutron fluence and irradiation temperature along the length of the thimble, shown in Fig. 2. Because of the small dimensions, only two specimens of a given irradiation temperature and fluence condition were obtained. By removing specimens from those segments of the thimble which were located below and above the reactor core, the effect of varying fast-neutron fluence at constant irradiation temperatures of 370 or 462°C was investigated.

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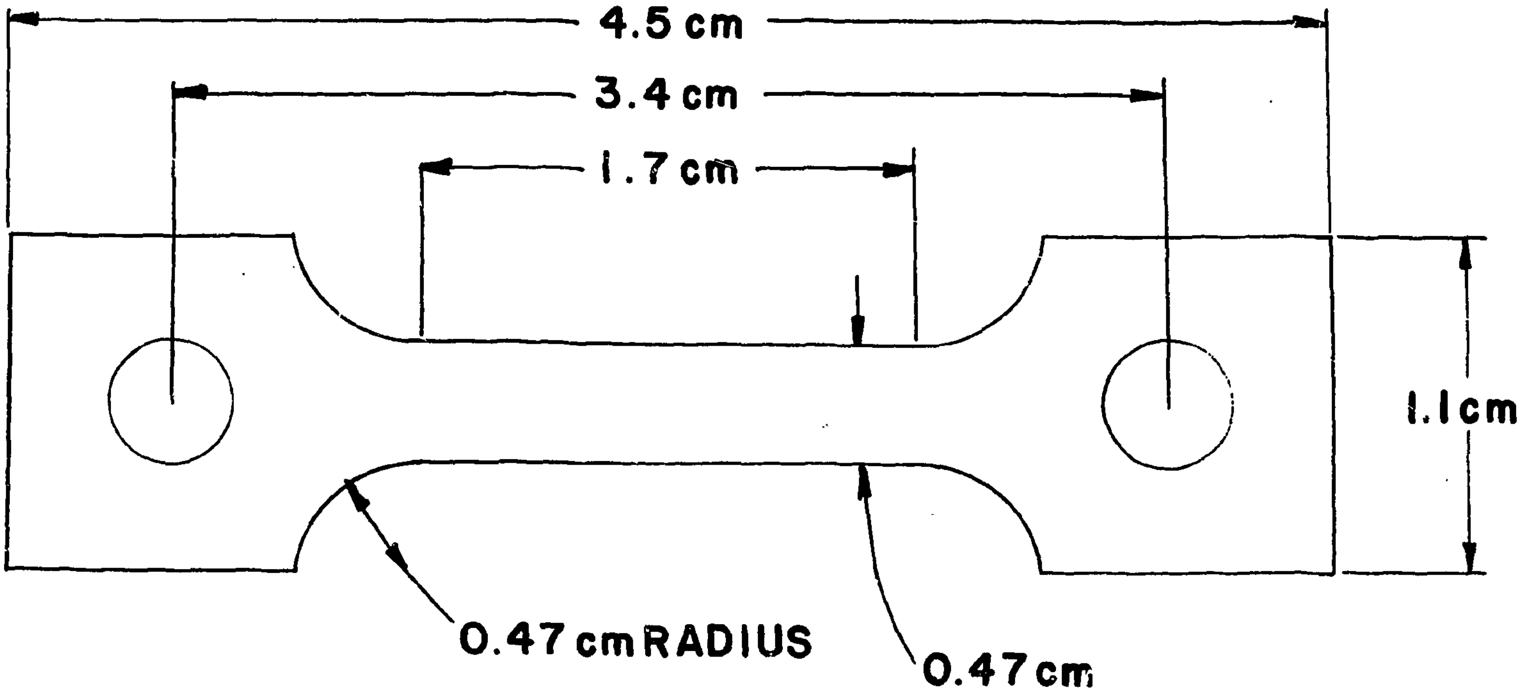


Fig. 1. Sheet Specimen Used for Postirradiation Creep-Rupture Testing of Experimental Breeder Reactor II Safety-Rod Thimble.

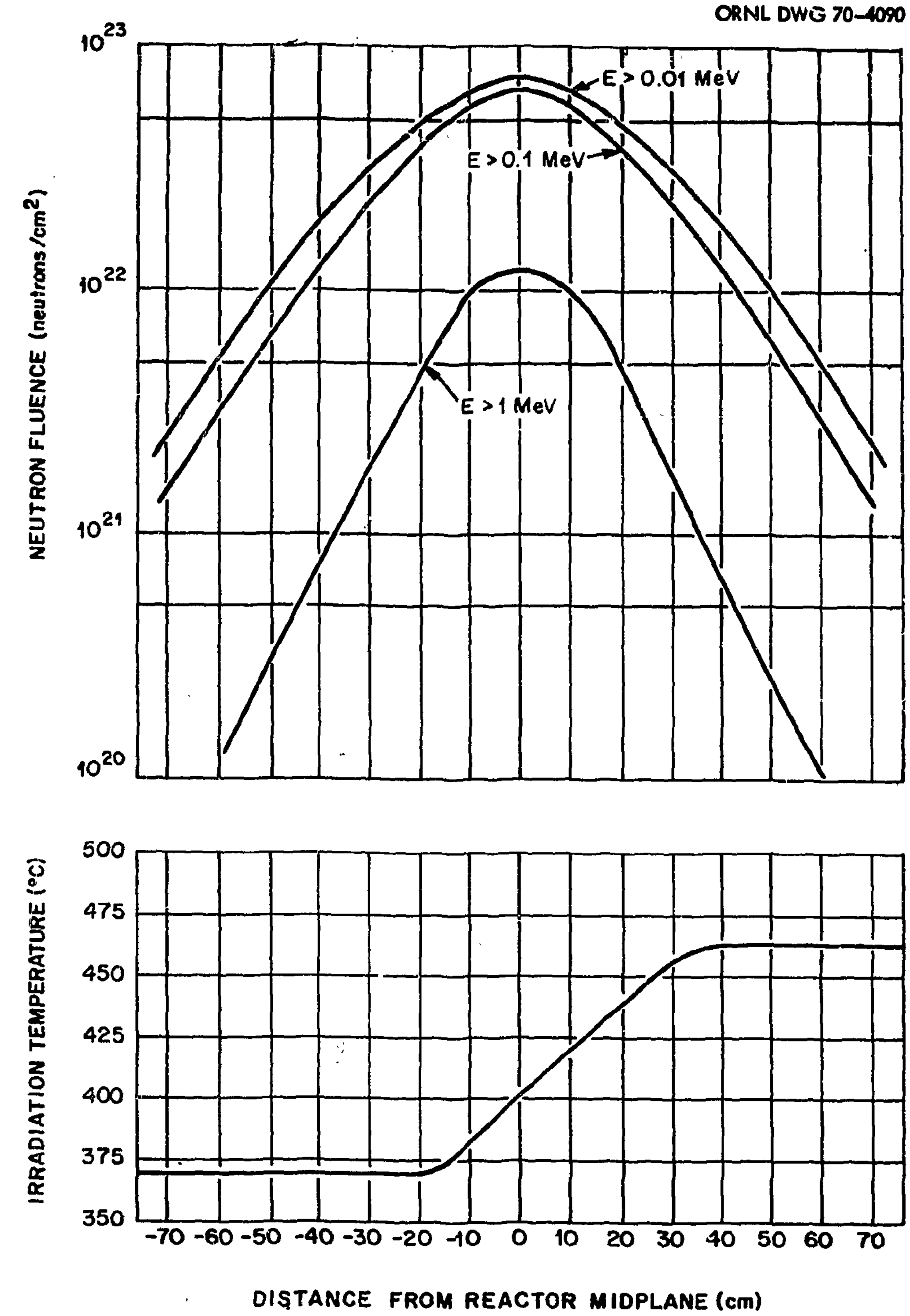


Fig. 2. Fast Neutron Fluence and Irradiation Temperature for Experimental Breeder Reactor-II Safety-Rod Thimble.

The microstructure of the material in the as-irradiated condition was characterized by transmission electron microscopy examination of specimens removed from various positions along the length of the thimble. The details of this portion of the experimental procedure have been discussed elsewhere.[13]

[13]E. E. Bloom, An Investigation of Fast Neutron Radiation Damage in an Austenitic Stainless Steel, ORNL-4580 (August 1970), Ph.D. Thesis The University of Tennessee.

In order to expand the range of irradiation temperature beyond that available through an examination of the safety-rod thimble, specimens of type 304 stainless steel were irradiated in an experimental subassembly located in row 2 of the reactor. The specimens were irradiated in specimen holders such as the one shown schematically in Fig. 3. A gas gap between the holder surface and the inside surface of the tube element provided a barrier to radial heat flow and allowed

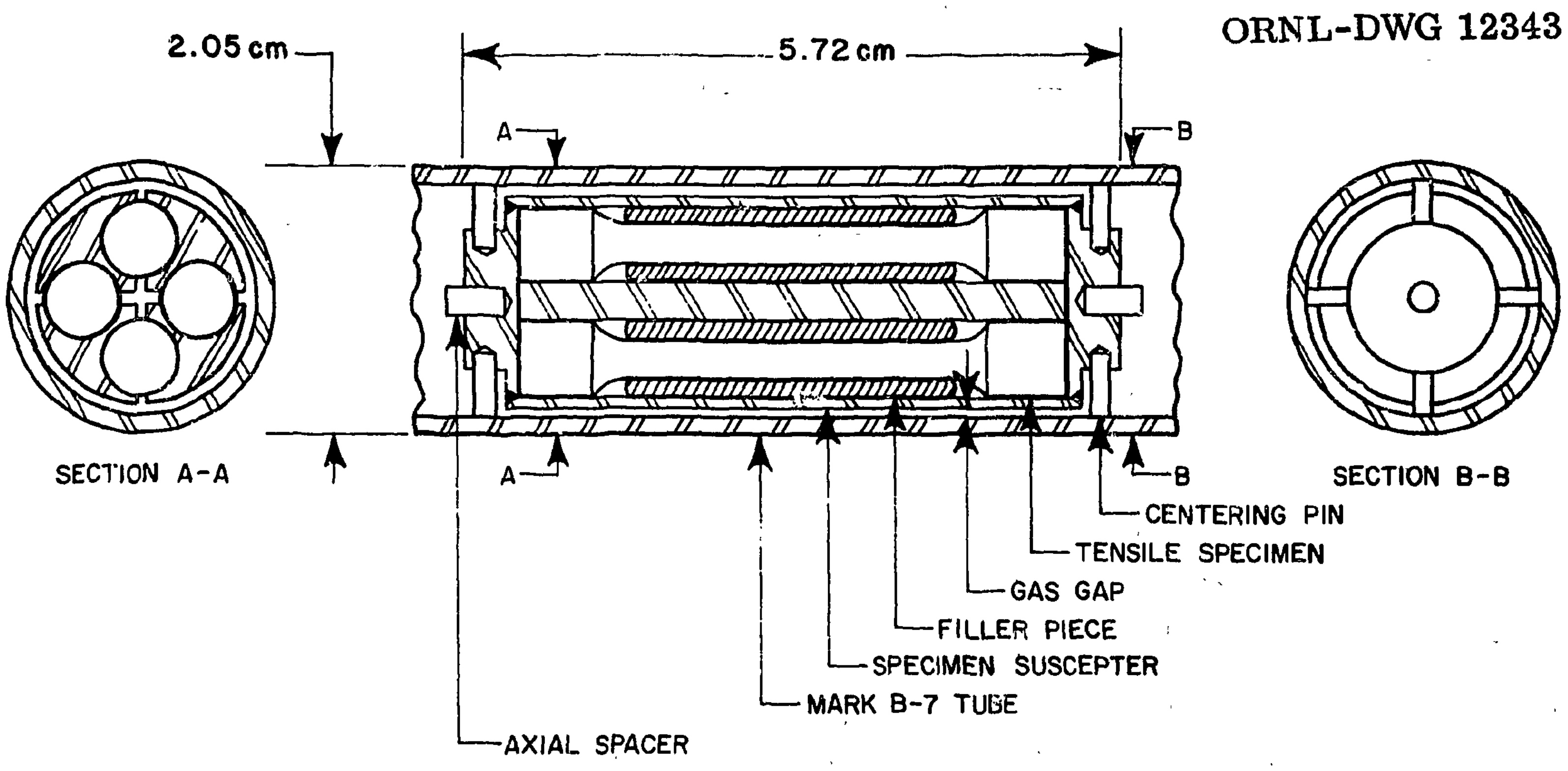


Fig. 3. Schematic Drawing of Specimen Holder Used in Experimental Breeder Reactor- II Materials Irradiation Experiments.

temperatures above the reactor-coolant temperature to be obtained. The experiment was designed and calibrated on the basis of nuclear heating rates as a function of position within the reactor as determined in a previous experiment. The calculated irradiation temperature was 600°C for a peak gamma heating rate of 3.5 w/g. Gamma heating rates of 3.0 and 4.0 w/g would give irradiation temperatures of 570 and 630°C, respectively.

Postirradiation creep-rupture tests were performed in air at 600 ± 5°C in lever-arm creep machines located in hot cells. 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. Creep-rupture tests on specimens machined from an unirradiated EBR-II safety-rod thimble were run in order to determine the change in properties due to irradiation. Examination of tested specimens included optical metallography, transmission microscopy, and scanning electron microscopy.

RESULTS

As-Irradiated Microstructures

Specimens irradiated to fast-neutron fluences in the range 2×10^{21} to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev) at temperatures between 370 and 470°C contained voids and faulted interstitial dislocation loops. The details of the microstructures have been reported elsewhere.[13] At low fast-neutron fluences [below 10^{22} neutrons/cm^2 (> 0.1 Mev)] the damage was heterogeneously distributed. Dislocation loops were clustered around grown-in dislocation lines, and voids were often located on dislocation lines. For specimens irradiated to a constant fast-neutron fluence, the void and dislocation loop concentrations decreased and the size of these defects increased with increasing temperature. Examples of the dislocation loop structure and void structure for specimens irradiated at 370 to 380 and 460 to 470°C are shown in Figs. 4 and 5. Regions on the order of 1000 A wide adjacent to grain boundaries were denuded of these defects. At fluences in excess of about 1×10^{22} neutrons/cm^2 (> 0.1 Mev) the loop concentration was so high that quantitative microscopy measurements were impossible. The void concentration increased monotonically as a function of fluence as shown in Fig. 6. After irradiation at 570 to 630°C to fluences of 2.5 to 3.5×10^{22} neutrons/cm^2 (> 0.1 Mev) the damage structure consisted of a dislocation network, a few unfaulted dislocation loops, and voids ranging up to about 600 A in diameter. A typical microstructure is shown in Fig. 7. The dark, rectangular particle is a thin sheet precipitate.

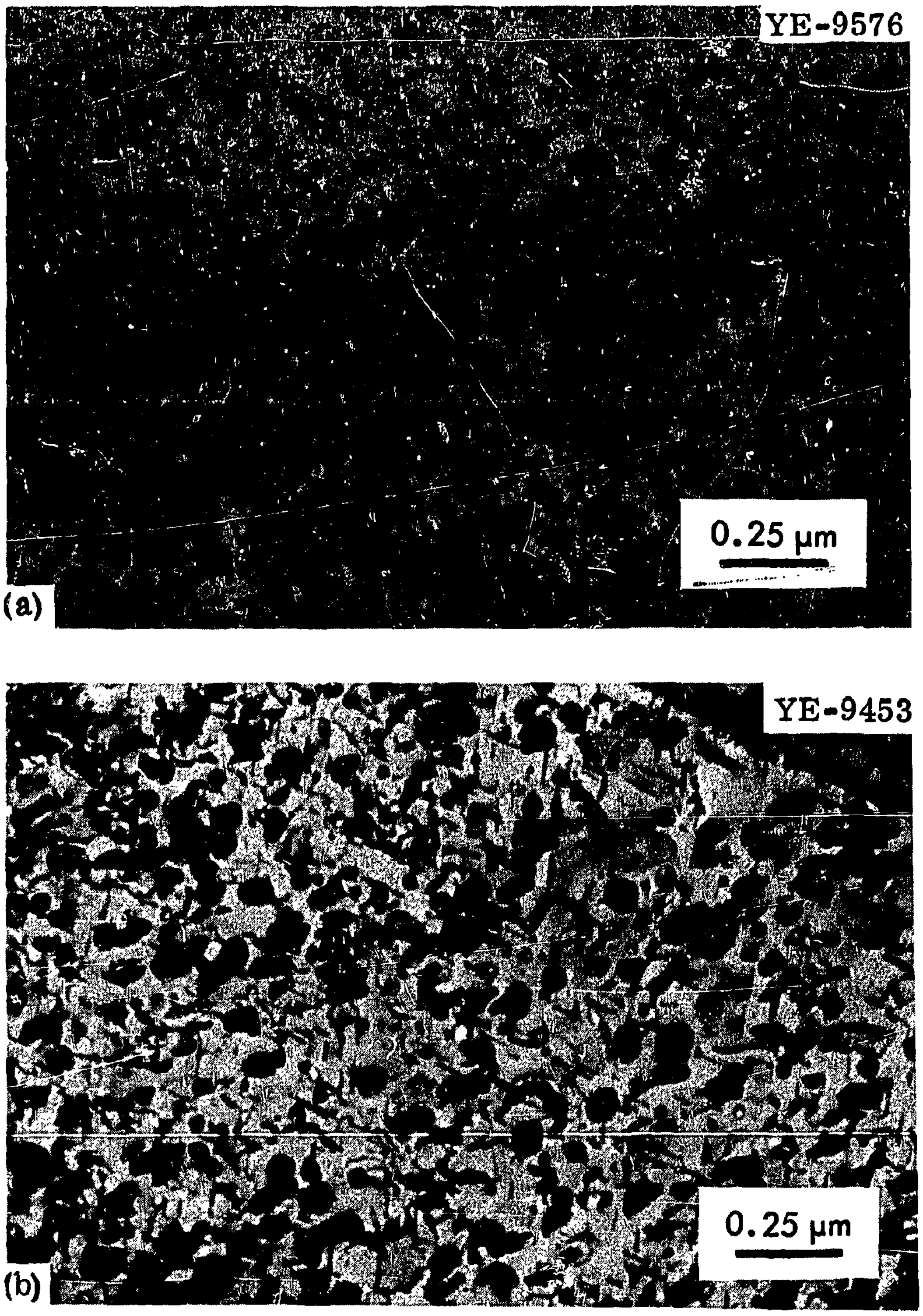
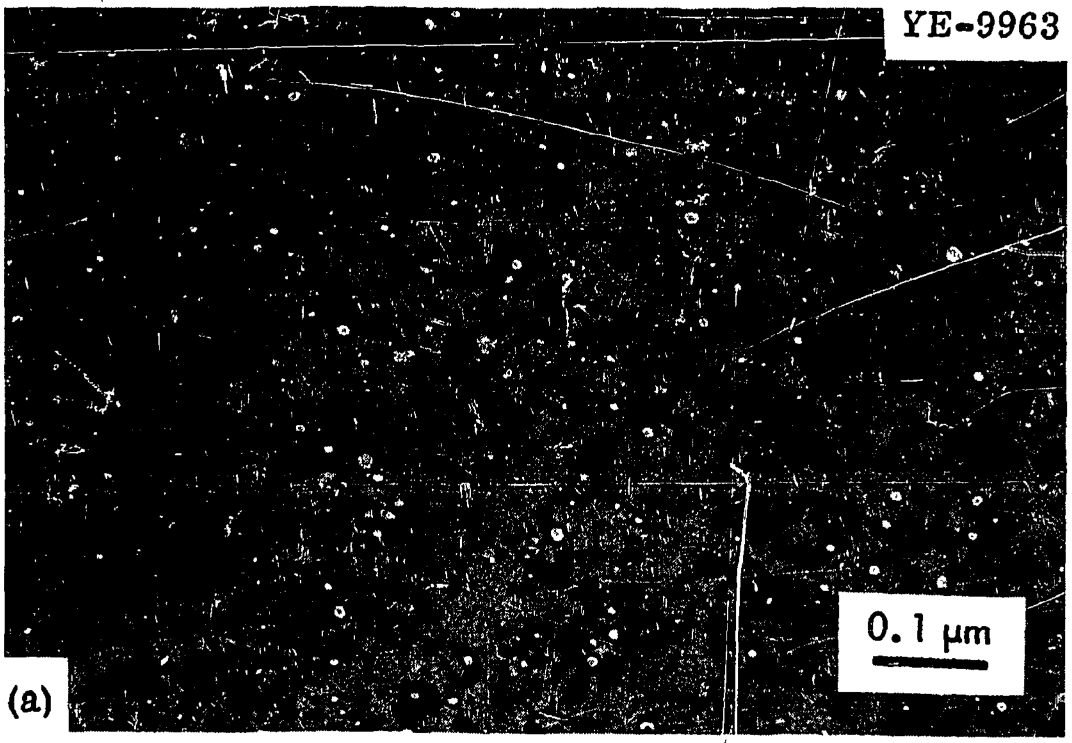


Fig. 4. Dislocation Loops in Type 304 Stainless Steel Irradiated at (a) 370°C to 0.8×10^{22} neutrons/cm^2 (> 0.1 Mev) and (b) 460°C to 0.9×10^{22} neutrons/cm^2 (> 0.1 Mev).



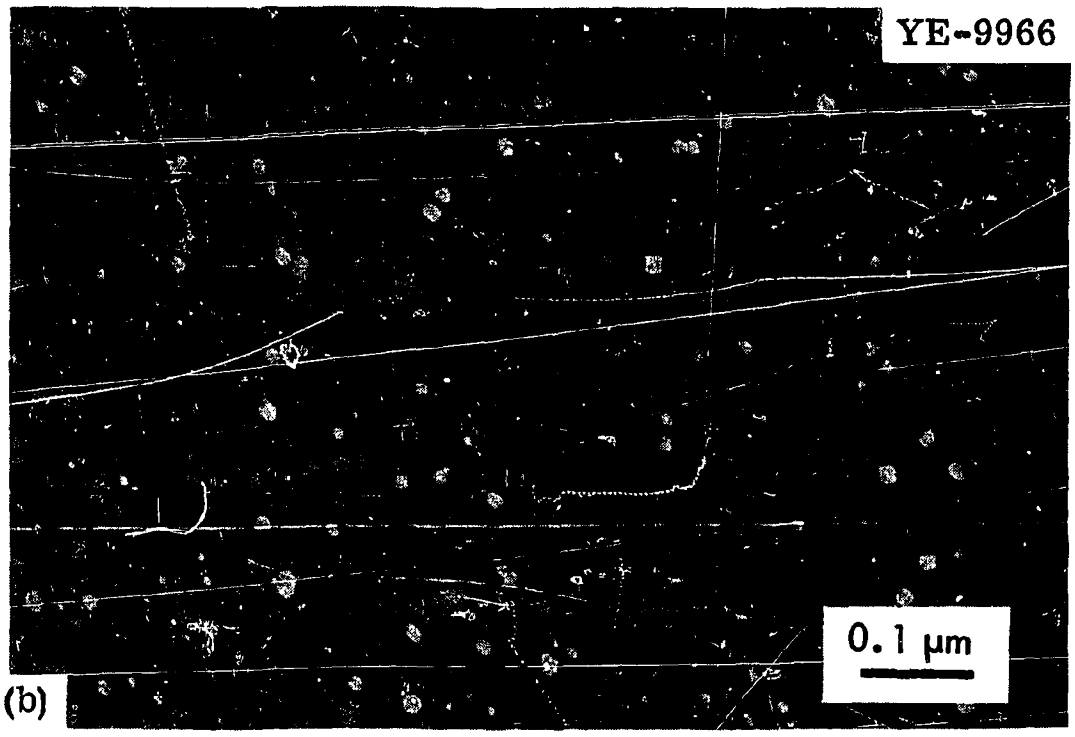


Fig. 5. Voids in Type 304 Stainless Steel Irradiated at (a) 370°C to 1.2×10^{22} neutrons/cm^2 (> 0.1 Mev) and (b) 460°C to 2.1×10^{22} neutrons/cm^2 (> 0.1 Mev).

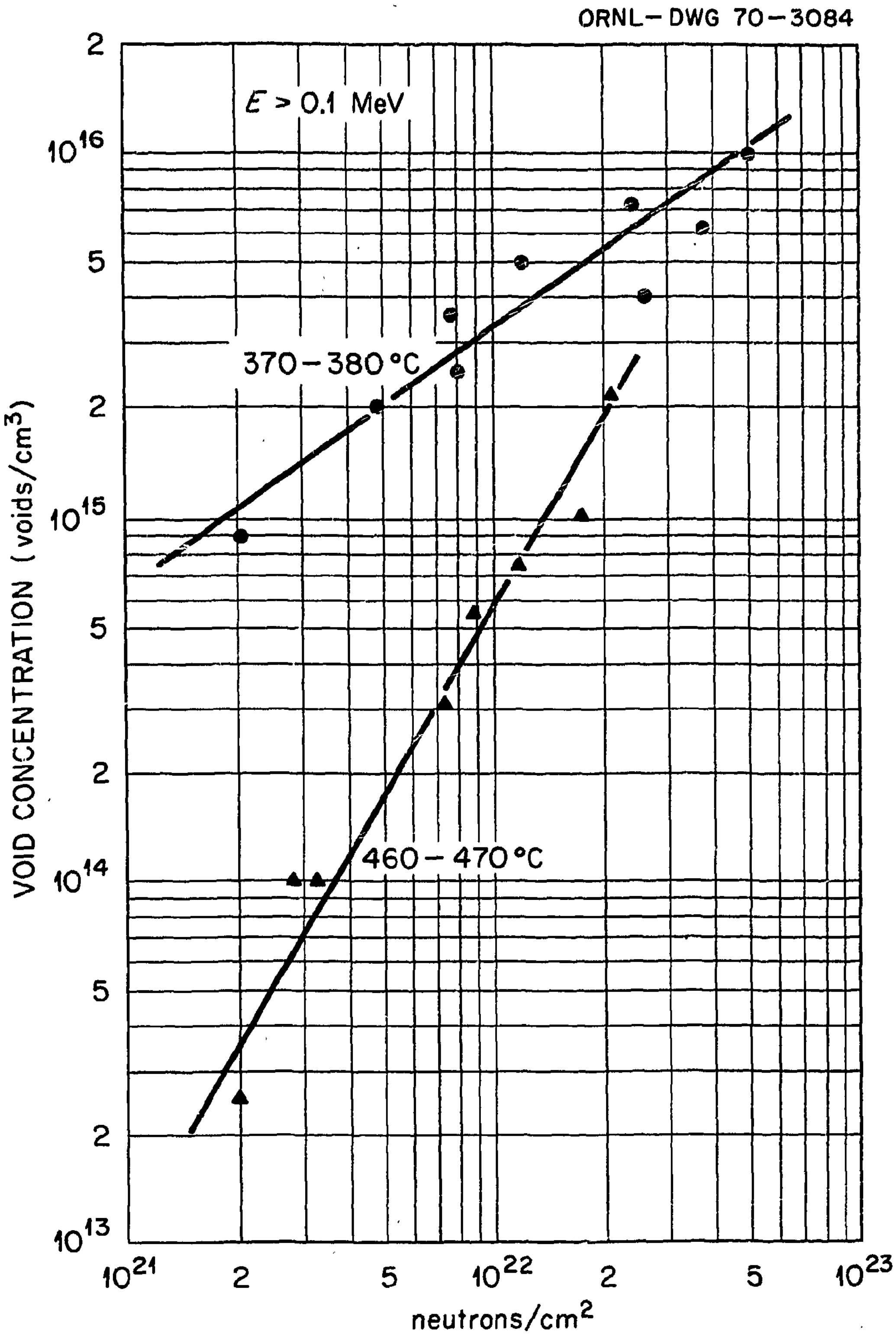


Fig. 6. Variation of Void Concentration with Fast-Neutron Fluence in Type 304 Stainless Steel Irradiated at 370 to 380 and 460 to 470 °C.

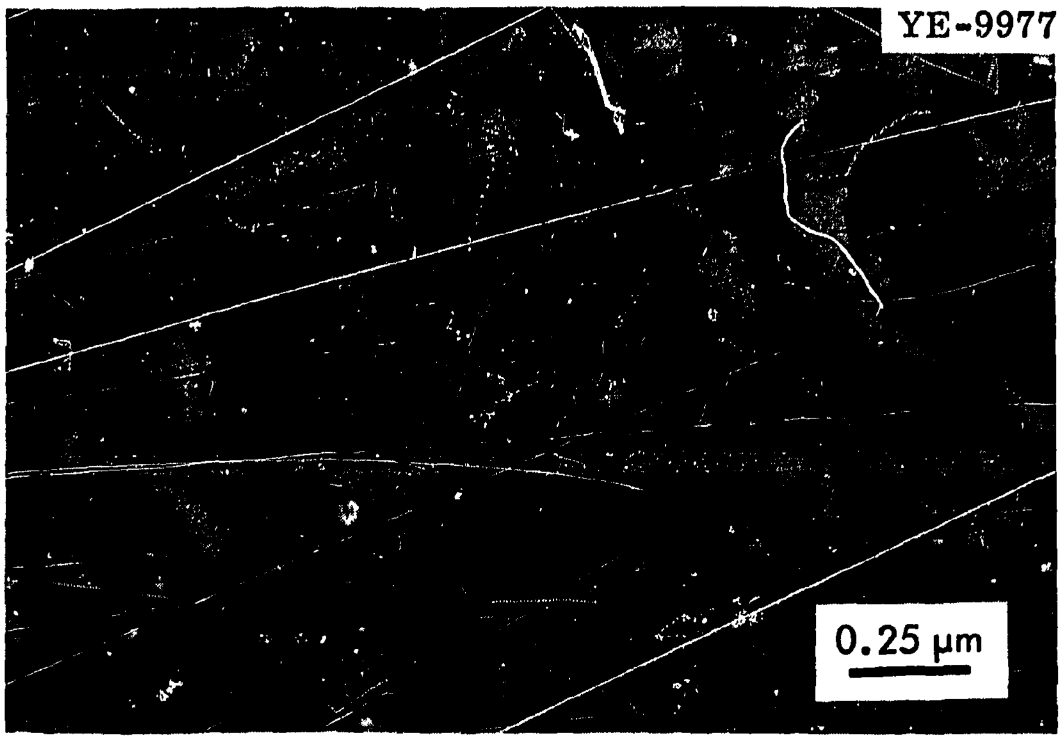


Fig. 7. Dislocation Network and Voids in Type 304 Stainless Steel Irradiated at 600°C to 3.5×10^{22} neutrons/cm^2 (> 0.1 Mev).

Creep-Rupture Properties

The creep-rupture properties at 600°C of sheet specimens, removed from an unirradiated safety-rod thimble and of rod specimens of the same heat as was irradiated in the experimental subassembly (heat BO), are shown in Fig. 8. The strength properties of these two heats of type 304 stainless steel are typical for this alloy, and the variation in properties between these two heats is well within the heat-to-heat variation as discussed by Smith.[14] Ductility, as measured by total elongation, ranged from 16 to 40% for specimens of the safety-rod thimble and from 9 to 25% for specimens from heat BO.

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[14]G. V. Smith, An Evaluation of the Yield, Tensile, Creep and Rupture Strengths of Wrought 304, 316, 321, and 347 Stainless Steels at Elevated Temperatures, ASTM DS 552, American Society For Testing and Materials, Philadelphia, 1969.

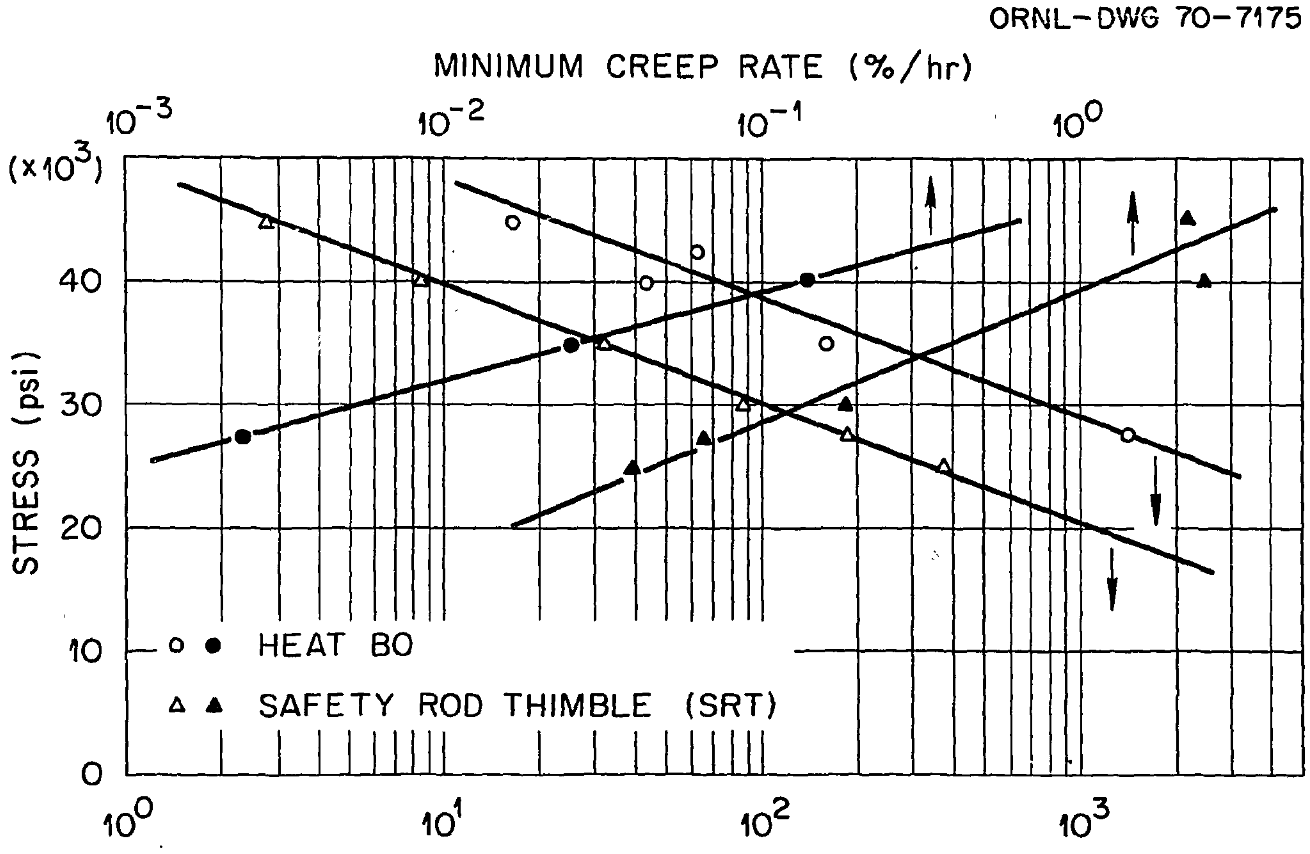

Fig. 8. Creep-Rupture Properties of Two Heats of Type 304 Stainless Steel at 600°C.

The effect of fast-neutron fluence on the rupture life of specimens of the safety-rod thimble which were irradiated at temperatures in the range 370 to 460°C and tested at 600°C and 27,500 psi is shown in Fig. 9. The rupture life of the unirradiated safety-rod thimble at this temperature and stress was 185 hr. Specimens irradiated at 370°C exhibited shorter rupture lives than did specimens irradiated at 460°C. For each irradiation temperature, the rupture life decreased sharply with increasing fast-neutron fluence. A specimen irradiated at 410°C to about 6.5×10^{22} neutrons/cm^2 (> 0.1 Mev) had a rupture life of 0.55 hr, a factor of 330 less than the unirradiated material. The total elongation as a function of fluence for these specimens is shown in Fig. 10. The total elongation was reduced from values in the range of 15 to 25% for unirradiated specimens tested at these conditions to values in the range of 2 to 3% for specimens irradiated to fluences of 4×10^{21} neutrons/cm^2 (> 0.1 Mev). The ductility decreased continuously with increasing fast-neutron fluence and was essentially independent of irradiation temperature over the range 370 to 460°C.

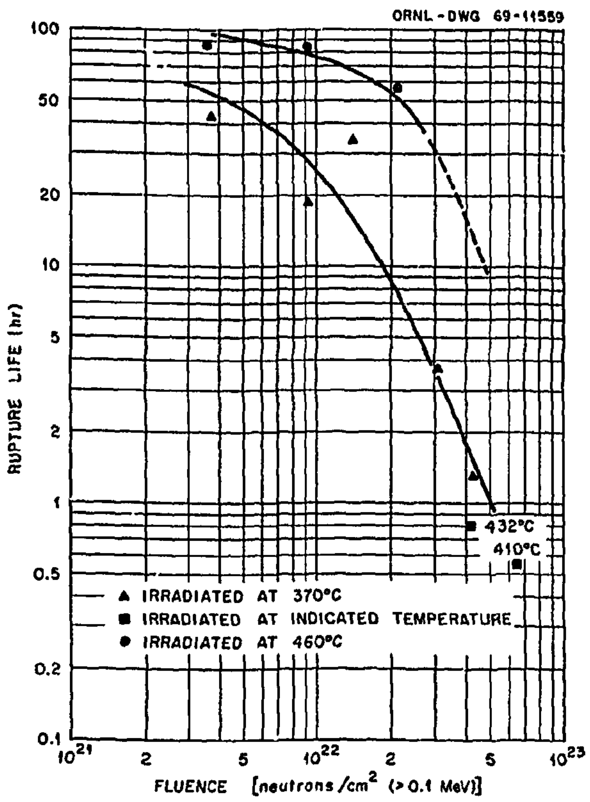

Fig. 9. Postirradiation Rupture Life of Type 304 Stainless Steel at 600°C and 27,500 psi. The rupture life of an unirradiated specimen tested at this temperature and stress was 185 hr.

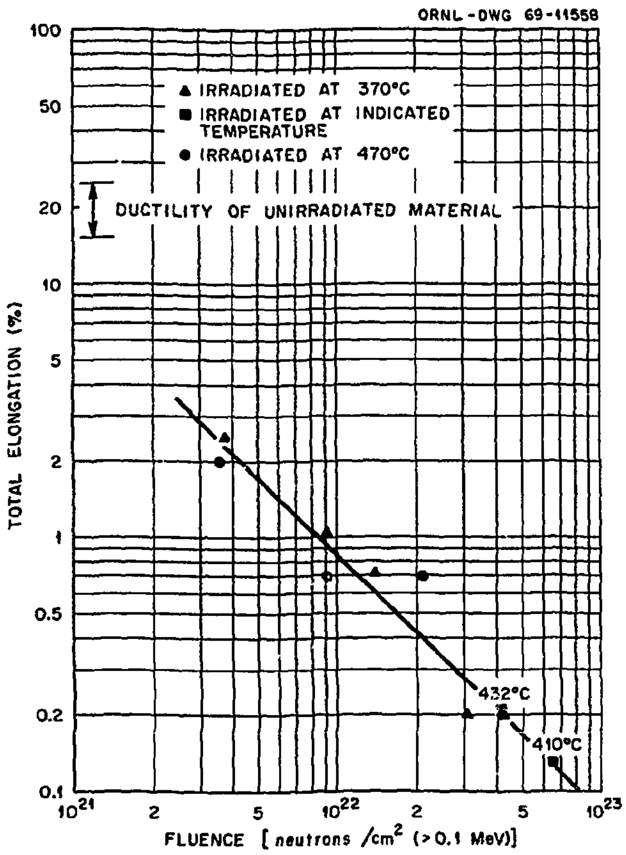

Fig. 10. Postirradiation Ductility of Type 304 Stainless Steel Tested at 600°C and 27,500 psi.

Typical strain-time curves for specimens irradiated at 370°C are shown in Fig. 11. Irradiated specimens exhibited a very short primary creep stage, usually less than 1 hr, as compared to 12 hr for an unirradiated specimen. Secondary creep rates were reduced by the irradiation, but for specimens irradiated to fluences greater than about 1×10^{22} neutrons/cm^2 (> 0.1 Mev) fracture occurred in such short times that the creep rates have little significance. None of the irradiated specimens exhibited tertiary creep.

Creep-rupture properties of specimens irradiated at 600°C to fast-neutron fluences in the range 2.5 to 3.5×10^{22} neutrons/cm^2 (> 0.1 Mev) are shown in Fig. 12. For these irradiation conditions, little or no change in the rupture life was observed; however, the ductility was significantly reduced. For example, an unirradiated specimen tested at 27,500 psi exhibited about 5% elongation at the onset of tertiary creep and about 9% total elongation at fracture as compared to an irradiated specimen tested at the same stress which exhibited 2.1% total elongation with no tertiary creep. Minimum creep rates of irradiated specimens were about a factor of 4 less than the unirradiated material. The net result of the reduced minimum creep rates and reduced ductilities was that irradiation did not change the rupture life.

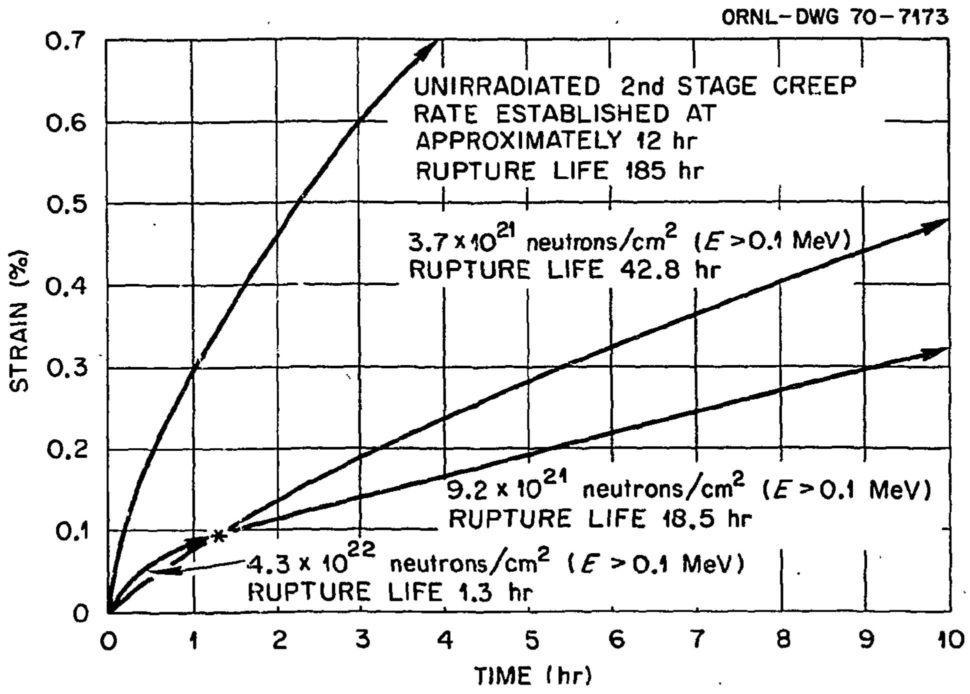

Fig. 11. Effect of Irradiation at 370°C on the Creep Curves of Type 304 Stainless Steel Tested at 600°C and 27,500 psi.

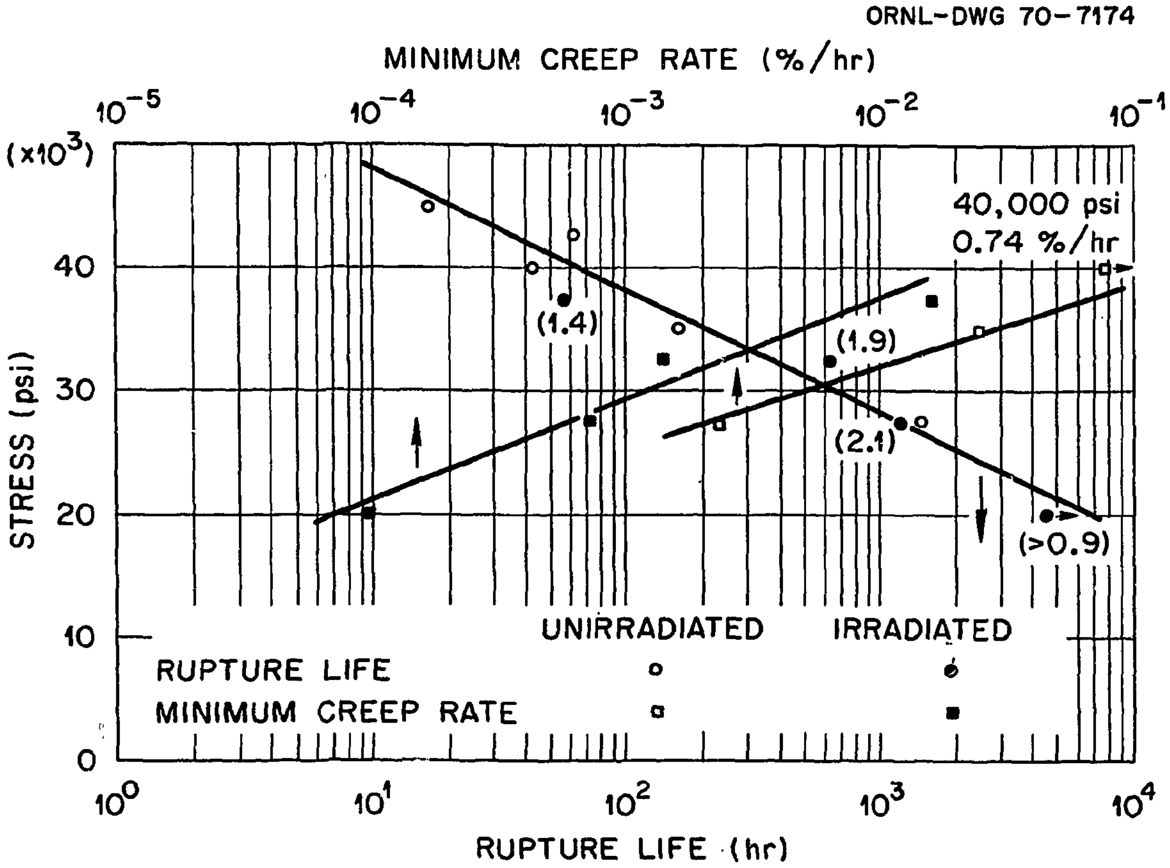

Fig. 12. Effect of Irradiation at 600°C to 2.5 to 3.5 × 10^{22} neutrons/cm^2 (> 0.1 Mev) on the Creep-Rupture Properties of Type 304 Stainless Steel at 600°C.

Metallography of Test Specimens

The fractures of all irradiated specimens were intergranular. Figure 13 shows a scanning electron micrograph of a specimen irradiated at 410°C to 6.7 × 10^{22} neutrons/cm^2 (> 0.1 Mev) and tested at 600°C and 27,500 psi stress. The intergranular fracture was initiated on the left-hand side of the specimen and propagated to the right. The right-hand portion failed last in a shear mode, presumably at a very high stress level and high strain rates. Optical metallography confirmed that even in those specimens irradiated to relatively low fluences the fractures were intergranular with no evidence of deformation within the matrix and little or no grain-boundary cracking in the region adjacent to the fracture. It thus appears that a grain-boundary crack, once initiated, propagates rapidly to cause failure.

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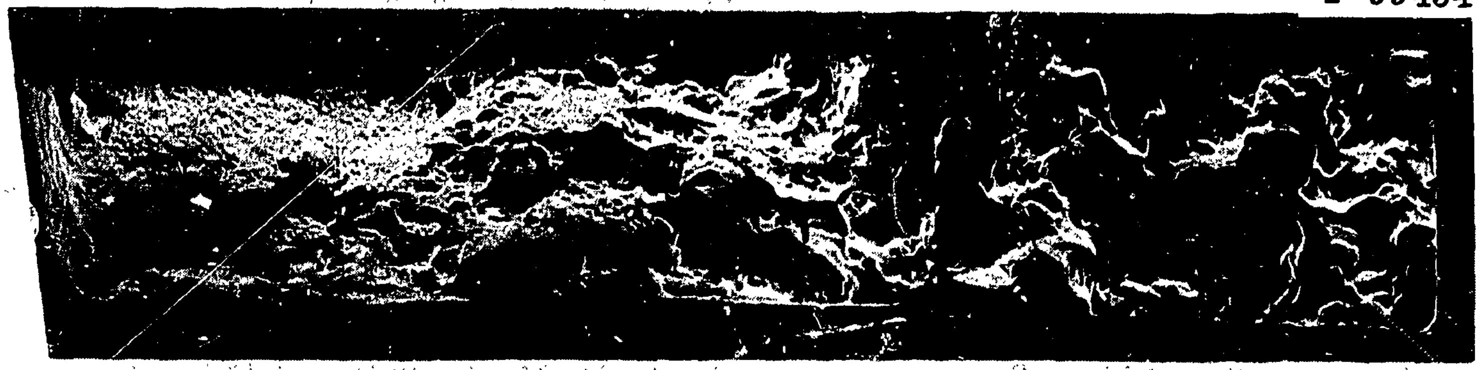

Fig. 13. Fracture of Type 304 Stainless Steel Irradiated at 410°C to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev) and Tested at 600°C and 27,500 psi.

For those specimens which were removed from the EBR-II safety-rod thimble the irradiation temperature was lower than the test temperature. It was thus important to determine the changes in microstructure which occurred during the test. Figure 14 shows a transmission micrograph of the area near the fracture of the same specimen as shown in Fig. 13. The specimen had been at 600°C for approximately 4 hr before failure occurred. Prior to testing, the structure consisted of voids and faulted dislocation loops. During the test, the dislocation loops unfaulted to produce the dislocation structure shown in Fig. 14. Previous results[15] have shown that unfaulting of the dislocation loops can occur during annealing 1 hr at 600°C. It is thus probable that the dislocation loops had unfaulted during the 3-hr hold time at 600°C prior to application of the stress. The dislocation density and configurations were very similar to those in a specimen irradiated under the same conditions and then annealed for 2 hr at 600°C. The lack of dislocation tangles and the similarity of microstructures between stressed and unstressed specimens are evidence that little deformation occurred within the matrix. The void concentration and size distribution in the tested specimen were approximately the same as in the as-irradiated condition.

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[15]J. O. Stiegler and E. E. Bloom, "The Effects of Large Fast-Neutron Fluences on the Structure of Stainless Steel," J. Nucl. Mater. 33, 173 (1969).

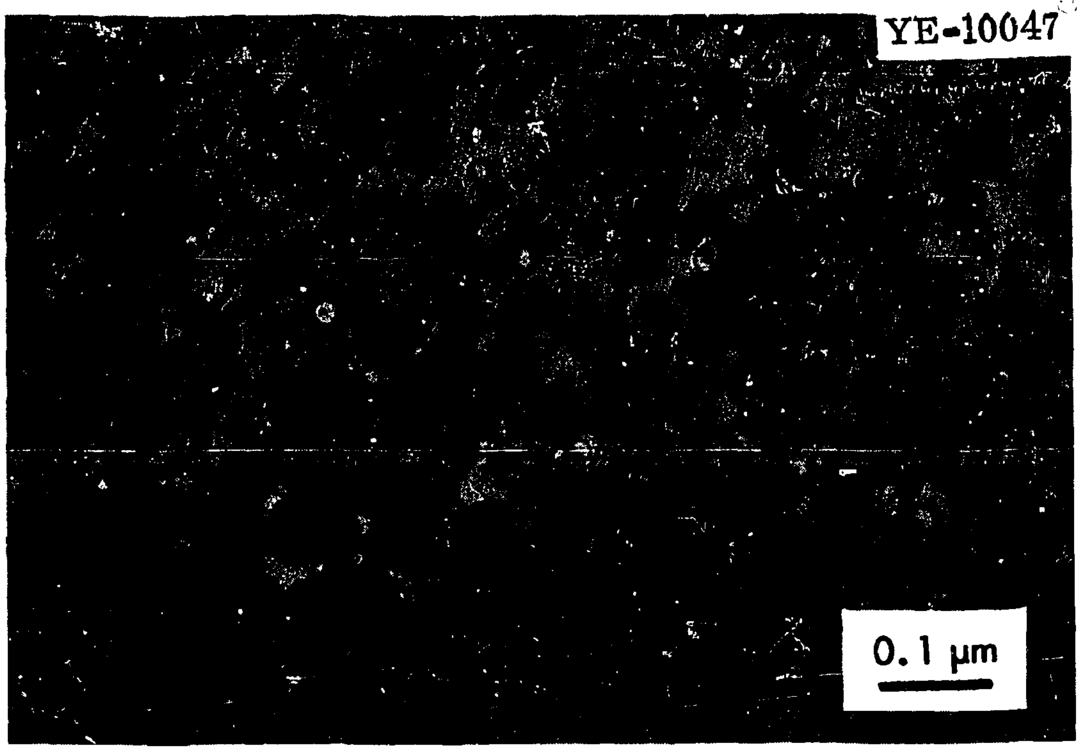

Fig. 14. Dislocation Structure to Type 304 Stainless Steel Irradiated at 410°C to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev) and Tested at 600°C and 27,500 psi.

SUMMARY OF RESULTS AND DISCUSSION

Specimens of type 304 stainless steel were irradiated at temperatures in the range 370 to 600°C to fast-neutron fluences in the range 2×10^{21} to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev) and then creep-rupture tested at 600°C. For irradiation at temperatures from 370 to 470°C, the as-irradiated microstructure consisted of voids and faulted interstitial dislocation loops. The void concentration increased with increasing fluence at constant irradiation temperature and decreased with increasing irradiation temperature at constant fluence.

Characterization of the dislocation loop structure in a quantitative manner was impossible due to the complexity and density of the structure. It was apparent, however, that the loop concentration decreased and the loop size increased with increasing irradiation temperature. Specimens irradiated at 600°C contained a dislocation network, a few unfaulted dislocation loops, and voids which were nearly always associated with either dislocations or precipitate particles.

The rupture life, ductility, and creep rate were all affected by the irradiation. For a given fast-neutron fluence, the reduction in rupture life was larger for specimens irradiated at 370°C than for specimens irradiated at 460°C. For these irradiation conditions, the reduction in rupture life is a result of the increased tendency of the material to fracture along the grain boundaries. Scanning electron microscopy of the fracture surfaces and optical metallography of the gage section of tested specimens indicated that the fractures were intergranular and that there were essentially no grain-boundary cracks along the gage section. It thus appears that a crack, once initiated, propagates very rapidly to cause failure. It is well documented[16–18] that the helium produced during the irradiation causes a reduction in ductility and rupture life due to its effects on grain-boundary fracture processes. The specimen which was irradiated at 410°C to 6.7×10^{22} neutrons/cm^2 (> 0.1 Mev) contained about 12 ppm He (ref. 19). This specimen exhibited about 0.1% total elongation and ruptured in 0.5 hr, a factor of 330 less than the unirradiated material. King[18] has shown that a uniform concentration of 20 ppm He reduced the rupture life of annealed type 304 stainless steel from 900 to 40 hr at 600°C and 30,000 psi. The reduction in rupture life and ductility of the fast-neutron-irradiated specimens may not be entirely due to the irradiation-produced helium. The intergranular fracture which is

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[16] D. R. Harries, "Neutron Irradiation Embrittlement of Austenitic Stainless Steels and Nickel Base Alloys," J. Brit. Nucl. Soc. **5**, 74 (1966).

[17] 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.

[18] R. T. King, "Cyclotron Simulation of Neutron Transmutation Produced Gases in Reactor Cladding and Structural Materials," paper presented at the International Conference on the Use of Cyclotrons in Chemistry, Metallurgy, and Biology, held at Oxford, England, September 1969 (to be published in the proceedings).

[19] Helium analysis was conducted by H. Farrar, Atomics International, Canoga Park, Calif.

observed at elevated temperatures is initiated by grain-boundary sliding.[20] In the irradiated specimens the regions adjacent to the grain boundaries are 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 a jogs and grain-boundary junctions. In an unirradiated specimen, 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.

In specimens irradiated at 600°C to 2 to 3×10^{22} neutrons/cm^2 the concentration of irradiation-produced voids and dislocations was much lower than in the specimens irradiated at lower temperatures; thus, some deformation within the matrix (and relaxation of stresses) could occur. The observation of no effect of irradiation on the rupture life in these specimens was a consequence of the reduced creep rate and reduced ductility. If the damage mechanism does depend upon the concentration of defects present in the matrix, the rupture life of specimens irradiated at 600°C and tested at 600°C would be expected to exhibit a fluence dependence similar to that for specimens irradiated at lower temperatures. This curve would be displaced to higher fluences because of the higher fluences required to establish a given defect concentration.

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[20] N. J. Grant, "Intercrystalline Failure at High Temperatures," pp. 562–578 in Fracture, ed. by B. L. Averbach, D. K. Felbeck, G. T. Hahn, and D. A. Thomas, Massachusetts Institute of Technology and Wiley, 1959.