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EFFECT OF NEUTRON IRRADIATION ON THE DUCTILITY
OF AUSTENITIC STAINLESS STEEL*

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17 Pages
1 Table
7 Figures

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ABSTRACT

The effects of fast neutron irradiation on the tensile and creep-rupture ductility of austenitic stainless steels are presented. At low temperatures, below 450°C, radiation causes a reduction in the strain hardening exponent and thus the uniform strain. At high temperatures (above 750°C) reduced ductility results from helium embrittlement. At intermediate temperatures, hardening and helium effects act synergistically to produce lower ductility than would result from either effect alone. Possible mechanisms responsible for the observed changes are discussed.

INTRODUCTION

Ductility, the ability of a material to deform without fracturing, is only infrequently used in a quantitative sense by the structural designer. The use of austenitic stainless steels for fuel cladding and core structural materials in Liquid Metal Fast Breeder Reactors (LMFBR) will require these alloys to operate at temperatures and stresses which produce plastic strain. Neutron irradiation causes a reduction of most ductility parameters. It is probable that strain, e.g., fracture strain or ductility, will be the design-limiting factor for many reactor components. The fast reactor fuel pin is an example: The failure criterion postulated in the fuel pin performance model of Boltax et al. (1) involves failure when the thermal creep strain in the clad reaches a fixed limit. The need for examining the effects of irradiation on ductility for conditions where the irradiation and test temperatures are similar is thus apparent. Analyses and predictions of the behavior of irradiated components under transient conditions are also important, since an irradiated material may be subjected to temperatures and stresses which are substantially different from those which exist during normal or steady-state operation. It is thus necessary to examine the effects of irradiation on ductility at temperatures different from the irradiation temperature.

This paper presents the effects of irradiation on the tensile and creep-rupture ductility of austenitic stainless steels, and discusses the mechanisms responsible for the observed changes. Understanding these mechanisms should improve our confidence in extrapolating and interpolating empirical correlations of existing data into regions where little or no data exist.

EFFECTS OF IRRADIATION ON TENSILE DUCTILITY

The effects of irradiation to fluence in the range 1.5 to 3.5×10^{22} neutrons/cm² (> 0.1 MeV) on the tensile properties of annealed type 304 stainless steel when irradiated and tested at the same temperature are shown as a function of temperature in Fig. 1. The increased yield stress at the lower irradiation temperatures has been discussed and related to the irradiation-produced defects (voids and dislocations) by several investigators (2), (3), (4).

A major factor affecting ductility at the lower temperatures is that the ultimate tensile stress does not increase as much as the yield stress. True stress-true strain curves for four temperatures are shown in Fig. 2. The behavior of the irradiated material over the entire plastic strain range and that of the unirradiated material at strains above about 1 to 2% can be approximated as:

$$\bar{\sigma} = \kappa(\bar{\epsilon})^\eta \quad (1)$$

where $\bar{\sigma}$ is the true stress, $\bar{\epsilon}$ the true strain, η the strain-hardening exponent, and κ is a constant. For this deformation law, plastic instability (or the end of uniform strain and the onset of necking) occurs when $\bar{\epsilon} = \eta$. Since irradiation at the lower temperatures (e.g., 450 and 550°C in Fig. 2) decreases η , the uniform strain decreases as a consequence. Irradiation produces hardening (an increase in yield stress) by virtue of the fact that the microstructural damage produced during irradiation (voids, dislocations, and precipitates) increases the force which must be applied to a dislocation to move it through the matrix. The decrease in strain-hardening exponent at high fluences is associated

with the fact that once the yield stress has been exceeded and deformation initiated, the deformation is confined to bands or channels (3) as shown in Fig. 3. The passage of the first few slip dislocations weakens the barriers within the bands, and subsequent deformation within the bands occurs with little strain hardening. The damage responsible for this behavior is expected to saturate with increasing fluence. Bullough et al. (5) and Harkness (6) have speculated that the swelling ceases either by the attraction of the irradiation produced dislocations to void surfaces by image forces; or at a time when the voids become interconnected by the dislocations. In either case, the accumulation of additional lattice defects and thus the mechanical property changes would cease. Recent microstructural investigations show that a saturation in the concentration of voids does occur in annealed type 304 stainless steel (7). Tensile results (8), (9) for annealed type 304 stainless steel irradiated to fluences up to about 5×10^{22} neutrons/cm² (> 0.1 MeV) indicate that the uniform strain for specimens irradiated and tested at 450°C decreases to a value of about 2%. The trends in the data indicate a saturation of the property change near this value.

A second ductility parameter, the reduction of area, is also reduced by irradiation. This implies either that the fracture mechanism is changed or that the initiation of the fracture occurs at lower total strains. Specimens irradiated to about 2.5×10^{22} neutrons/cm² (> 0.1 MeV) and tested at 450°C failed in a ductile, transgranular mode. The fracture shown in Fig. 3 was similar in appearance to those of unirradiated specimens. In unirradiated specimens tested at low temperatures (e.g., 450°C), the fractures probably initiate in a manner

similar to that proposed by Rogers (10) and Puttick (11). Cavities initially form in regions of high stresses caused by inhomogeneous deformation or at precipitate particles due to inhomogeneous strain at the matrix-precipitate interface. As straining proceeds, the cavities grow and eventually link 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. Figure 3 shows that near the tensile fracture of an irradiated specimen, cavities have formed at points where the deformation bands are interrupted, that is, at precipitates, grain boundaries, and twin boundaries. Irradiation did not change the local fracture mechanism but caused fracture initiation at low strains by concentrating the strain in the deformation bands. Thus, cracks may initiate and propagate by the same process in irradiated as in unirradiated material, but produce lower overall strains. Additionally, it appears that important effects on the macroscopic fracture stress are produced by irradiation at 450 and 550°C (Table I). These effects are not associated with irradiation produced voids and dislocations (12) and the attendant reduction in strain hardening. We speculate that the precipitates produced during irradiation at 450 and 550°C produce an increased number of cavity nucleation sites. Fracture thus interrupts the deformation at lower macroscopic strains and stresses.

With increasing irradiation and test temperature, the yield stress increase becomes smaller and the strain-hardening exponents of irradiated and unirradiated specimens approach the same values. The ductility loss at the highest temperatures (e.g., 750°C) has the characteristics of high-temperature helium embrittlement (13); in particular,

the fracture mode changes from mixed intergranular and transgranular for unirradiated material to entirely intergranular following irradiation.

For temperatures intermediate between 450°C, where ductility losses occur primarily as a result of different stress-strain behavior, and the highest temperatures, where ductility losses are due primarily to helium embrittlement, the two mechanisms act synergistically. The results presented in Fig. 2 show that at intermediate temperatures the reduction in uniform strain is larger than can be accounted for in terms of reduced strain-hardening exponents. For example, for the specimen irradiated at 450°C, $\eta = 0.05$ and $\epsilon_{\text{uniform}} = 0.06$ in agreement with the observation that the uniform strain is controlled by the strain-hardening exponent within the limits of experimental error. However, at 600°C, $\eta = 0.18$ and $\epsilon_{\text{uniform}} = 0.10$, a value inconsistent with the strain-hardening exponent. Kramer et al. (14) have shown that 30 at. ppm He (about a factor of 10 higher helium content than in the Fig. 2 samples) has no significant effect on the uniform tensile elongation of annealed type 304 stainless steel at or below 650°C. The ductility loss is greater than that expected from hardening effects or helium effects when either is considered separately.

EFFECTS OF IRRADIATION ON CREEP-RUPTURE DUCTILITY

The effects of fast neutron irradiation on the creep-rupture properties of austenitic stainless steels have been studied for a narrow range of irradiation conditions compared to the range of conditions to be expected in LMFBRs: (1) properties of annealed type 304 and annealed and 20% cold-worked type 316 stainless steel have been investigated as

a function of temperature for fluences up to about 5×10^{22} neutrons/cm² (> 0.1 MeV); and (2) the effects of fluence have been investigated to about 1.7×10^{23} neutrons/cm², but only for annealed type 304 stainless steel irradiated at 370–460°C and then tested at higher temperatures (15)–(18). Nearly all of the creep-rupture properties (primary creep, secondary creep rate, rupture life, and ductility) are changed by irradiation. For irradiation temperatures at which a void and dislocation structure is formed, the amount of primary creep and the secondary creep rate are reduced. At higher temperatures, phase transformations (precipitation of carbides, sigma and other intermetallic phases), and/or recovery of cold-worked structures can cause increases in creep rate by as much as 1 or 2 orders of magnitude compared to the preirradiation condition. The rupture life is affected to various degrees, depending on the change in strength (creep rate) and ductility. The most important effect of irradiation is a reduction in creep-rupture ductility. The ductility after irradiation is strongly dependent on both the irradiation and test temperature as well as the fast neutron fluence.

When austenitic stainless steels are irradiated and tested at the same temperature, the reduction of ductility as measured either by strain at the beginning of tertiary creep or total elongation depends on both temperature and initial microstructure. The creep-rupture ductilities of annealed and 20% cold-worked type 316 stainless steel in the unirradiated condition and after irradiation to fluences in the range 1.2 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) are shown in Fig. 4. Irradiation of the annealed material caused a reduction in both the strain

at the beginning of tertiary creep and the total elongation at all temperatures. Irradiation reduced the ductility of 20% cold-worked material in the range 550 to 650°C while at 700 and 750°C increased ductility was observed. Metallographic examination of these samples (19) (reported in detail elsewhere) showed that the fractures were predominantly intergranular and occurred by the linking of cracks on grain boundaries which were approximately normal to the applied load. The cracks formed by the stress-induced growth and linking of grain boundary cavities. Phenomenologically, the temperature dependence of the ductility is associated with the extent to which cracks propagate, which in turn depends on the strength of the matrix. For example, irradiation of annealed type 316 stainless steel at the lower irradiation temperatures (550 and 600°C) caused relatively high concentrations of voids and dislocations. Cracks in the region of the fracture propagated for large distances, often across several triple grain junctions [Fig. 5(a)]. Well away from the fracture cracks had also formed, but these cracks remained very narrow, indicating that little grain-boundary sliding or matrix strain had occurred. In contrast, at 650°C where the concentration of irradiation-produced defects was considerably lower, cracks seldom extended across more than one or two triple grain junctions [Fig. 5(b)]. In addition, these cracks (even at locations far from the fracture) were wedge-shaped, indicating that deformation had occurred to open the crack. At the highest temperatures, nearly all of the boundaries contained either cracks or cavities [Fig. 5(c)]. Crack propagation and the percentage of intergranular fracture was greater for specimens irradiated in the cold-worked condition at 550 to 650°C.

than for the specimens irradiated in the annealed condition at 550 and 600°C. The low ductility in 20% cold-worked material irradiated at 650°C compared to the higher values for the annealed condition may be related to the fact that the cold-worked structure was not recrystallized during irradiation. As a result the matrix strength was higher than for the annealed material. Significant crack propagation occurred in the cold-worked specimens where, as in the annealed material, crack propagation appeared to be most difficult at this temperature. The higher ductility for specimens irradiated in the cold-worked condition at 700 and 750°C as compared to the annealed condition was associated with extensive recovery and recrystallization and may result from the fine grain size which developed (20).

The damage structure in the matrix and grain-boundary crack initiation and propagation appeared to be related in the following way. The stress-induced growth of grain-boundary cavities (21) and the linking of these cavities to form a crack occurred at all temperatures. Helium bubbles may have served as cavity nuclei. Once a crack had formed a stress concentration existed at its tip. For low irradiation and test temperatures (550 to 600°C for the annealed condition and 550 to 650°C for the 20% cold-worked condition), this stress concentration could not be relaxed due to the high flow stress of the matrix, and cavities near a crack tip were subjected to higher stresses and grew faster than cavities on an uncracked boundary. The cracks became self-propagating and caused failure before significant matrix deformation could occur. Intergranular fractures were observed under these conditions except over the last area to fail, in which case shear

fracture at a high strain rate occurred. At higher temperatures (650°C for the annealed specimens) cracks still formed by the stress-induced growth and linking of cavities. The cracks did not propagate, however, because the stress concentration in the vicinity of the crack tip was reduced by flow in the relatively soft surrounding matrix. Fracture occurred only when a large number of cracks formed and caused an increase in the stress due to the reduction in the effective cross-sectional area. Higher ductility is observed compared to lower temperatures because the time to fracture is sufficiently long compared to the creep strength to allow creep deformation to occur. Further increases of temperature increase the cavity growth rate by diffusion, again allowing the cavities to link and cause fracture before significant deformation occurs.

The importance of the irradiation-produced defect structure is further illustrated in Fig. 6 which shows strain-time curves for specimens of annealed type 304 stainless steel which were irradiated at temperatures of 450, 550, 600, and 780°C to a fluence of about 1.9×10^{22} neutrons/cm² and tested at a common stress and temperature of 35,000 psi and 550°C. The creep rate decreased sharply for the lower irradiation temperatures, consistent with the larger concentration of barriers (voids and dislocations). Corresponding to this decreased creep rate, there was a marked reduction in ductility. Since these samples were irradiated to about the same fluence, they contained similar amounts of helium. The differences in ductility cannot be attributed solely to helium embrittlement. Since the samples were tested at a common temperature and stress, the results indicate that the radiation-produced defect structure (i.e., voids, dislocations, and possibly precipitates) influences the properties.

An important aspect of the results shown in Fig. 6 is that large reductions in ductility and rupture life occur when a material is irradiated at a low temperature and then tested at a higher temperature. The total creep elongation of annealed type 304 stainless steel irradiated at 370 to 470°C and tested at 550 and 600°C at stresses of 35,000 and 27,500 psi, respectively, decreased sharply with increasing fluence (Fig. 7). Total elongations were about 0.1% at the higher fluences. Correspondingly large reductions in rupture life also occurred (from about 320 hr and 185 hr for the 550 and 600°C in the unirradiated condition to values of about 0.5 hr at the highest fluences). Although the fracture of these samples was intergranular, stress-induced growth of cavities may not be involved in this case. Indeed, the rupture times are much too short for this mechanism to be operative. Because only one crack was usually observed in the sample, that is, the fracture, it is suggested that once a crack initiated, it propagated by ductile tearing of the damage-denuded zone adjacent to the boundary. The high flow stress of the matrix and inability to relax stress concentration at the crack tip may also be important in this situation.

SUMMARY

Neutron irradiation produces significant changes in the physical and mechanical properties of austenitic stainless steels. Ductility is the most adversely affected property, and it is reduced for nearly all irradiation and test conditions. The fracture mechanisms responsible for and the magnitude of the ductility loss depend upon irradiation temperature, fast neutron fluence, initial microstructure, composition of the alloy, and test variables such as test temperature and strain rate.

For the relatively high strain rates, such as in tensile tests, irradiation and testing at 450°C and below causes reduced uniform strain as a result of the lower strain-hardening exponent and plastic instability at low true strain values. For temperatures of 750°C and above, the reduced ductility results from helium embrittlement. For intermediate temperatures, hardening and helium effects act synergistically to produce lower ductility than would result from either effect above. The low temperature effect appears to saturate at a uniform strain of 1 to 2%. Loss of ductility due to helium embrittlement is not expected to saturate since the helium content increases monotonically with fast fluence.

For low strain rate creep-rupture tests performed at the irradiation temperature, intergranular fractures and a severe loss of ductility occur in the range 550 to 650°C. Total elongations of 0.5 to 4.0% are observed at fluences of about 3×10^{22} neutrons/cm². The loss of ductility appears to be related to both the strength of the matrix and to helium content and thus should become more severe at higher fluences. In annealed specimens, the ductility increases with increasing temperature up to about 650°C due to the decreasing concentration of damage in the matrix. In cold-worked material, low ductilities are observed to somewhat higher temperatures, about 650°C, above which temperature recovery and recrystallization begin to occur and the ductility increases.

The loss of ductility is so severe that it must be considered in the analysis of both steady-state and transient operating conditions. The results on annealed and cold-worked type 316 stainless steel show that, of the temperatures of interest for steady-state operation in

LMFBRs, the range 550 to 650°C produces the most pronounced reduction in creep-rupture ductility. In this temperature range, no mechanism is apparent which would cause saturation of the effect. Indeed, in type 304 stainless steels irradiated to high fluences, total elongations as low as 0.1% have been observed.

In the case of a specimen which accumulates damage at a low temperature (e.g., 450°C) and then is stressed at a higher temperature (e.g., 550°C), a very severe loss of ductility and rupture life occur. This result must be considered in the analysis of behavior of components under transient conditions.

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TABLE I.

Effect of Irradiation on the True Fracture Stress and True Fracture Strain of Type 304 Stainless Steel.

Temperature (°C)	True Fracture Strain		True Fracture Stress	
	Unirradiated	Irradiated	Unirradiated	Irradiated
450	0.88	0.44	126,000	101,000
550	0.67	0.22	92,000	86,000
600	0.61	0.18	46,000	46,000

FIGURE CAPTIONS

Fig. 1. Effect of Irradiation on the Tensile Properties of Annealed, Type 304 Stainless Steel When Irradiated and Tested at Approximately the Same Temperature.

Fig. 2. Effect of Irradiation on the True Stress-True Strain Behavior of Annealed Type 304 Stainless Steel. Specimens were irradiated near the respective test temperatures to 1.5×10^{22} neutrons/cm² (> 0.1 MeV).

Fig. 3. Deformation Bands and Fracture in Type 304 Stainless Steel Irradiated in the Annealed Condition at 450°C to 2.5×10^{22} neutrons/cm² (> 0.1 MeV) and Tensile Tested at 450°C and 0.002 min⁻¹.

Fig 4. Effect of Fast Neutron Irradiation on the Creep-Rupture Ductility of Annealed and 20% Cold-Worked Type 316 Stainless Steel. Samples were irradiated to fluences in the range 1.2 to 2.7×10^{22} neutrons/cm². Irradiation and test temperatures were approximately the same.

Fig. 5. Creep-Rupture Fractures of Type 316 Stainless Steel Irradiated in the Annealed Condition and Tested at the Indicated Temperatures and Stresses.

Fig. 6. Effect of Fast Neutron Irradiation Temperature on the Creep Rupture of Annealed Type 304 Stainless Steel.

Fig. 7. Effect of Fast Neutron Fluence on the Creep-Rupture Elongation of Annealed Type 304 Stainless Steel at 550 and 600°C.

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Fig. 4. Effect of Fast Neutron Irradiation on the Creep-Rupture Ductility of Annealed and 20% Cold-Worked Type 316 Stainless Steel. Samples were irradiated to fluences in the range 1.2 to 2.7×10^{22} neutrons/cm². Irradiation and test temperatures were approximately the same.

(a) Irradiated 580°C, 2.7×10^{22} neutrons/cm² (> 0.1 MeV)
Tested 550°C, 45,000 psi

(b) Irradiated 630°C, 2.3×10^{22} neutrons/cm² (> 0.1 MeV)
Tested 650°C, 25,000 psi

(c) Irradiated 735 to 795°C, 2.7×10^{22} neutrons/cm² (> 0.1 MeV)
Tested 750°C, 7,500 psi

Fig. 5. Creep-Rupture Fractures of Type 316
Stainless Steel Irradiated in the Annealed Condition
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