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EFFECT OF IRRADIATION OF THE MICROSTRUCTURE AND CREEP-RUPTURE PROPERTIES
OF TYPE 316 STAINLESS STEEL*

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ABSTRACT

The effect of irradiation to fluences in the range 1.2 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) on the microstructure and creep-rupture properties of annealed and cold-worked type 316 stainless steel have been examined for irradiation temperatures in the range 450 to 825°C . For irradiation in the annealed condition below about 650°C , voids and dislocations were formed. Cold working before irradiation suppressed void formation at all temperatures except 580°C where voids were occasionally found in recovered areas. Extensive precipitation occurred at temperatures above about 500°C , and the amount and type of precipitate was sensitive to the initial microstructure. Changes in rupture life and creep rate were observed, but the most significant effect of irradiation was a large decrease in ductility resulting from enhanced intergranular fracture. The grain boundary cracks were formed by the linking of cavities on those boundaries nearly normal to the applied stress. The experimental results are most consistent with a model in which nucleation of the cavities occurs on helium bubbles with growth controlled by stress enhanced diffusion of vacancies. Experimental evidence also indicates that the strength of the matrix influences the crack propagation rate.

INTRODUCTION

Type 316 stainless steel will be used for fuel element cladding and fuel subassembly ducts in the FTR and possibly in future liquid metal fast breeder reactors. In this application the alloy will operate in the temperature range 350 to 750°C for times of several thousand hours. This alloy is unstable and such thermal exposure will cause the precipitation of carbides (primarily $M_{23}C_6$) and intermetallic phases such as sigma, chi, and eta or laves.¹ Voids and a dislocation structure will be formed by the precipitation of irradiation-produced vacancies and interstitials at temperatures in the range 350 to 650°C, and helium bubbles will be formed at higher irradiation temperatures.^{2,3} In order to reduce the swelling caused by the void-dislocation structure, the alloy will probably be used in the cold-worked condition^{4,5}, but a cold-worked structure will significantly alter the kinetics of the carbide and sigma precipitation reactions, in general by causing the time-temperature-precipitation curves to be shifted to shorter times or lower temperatures. In addition there is some evidence that the precipitation kinetics and phase stability may be altered by irradiation.⁶

These complex microstructural changes may have a pronounced effect on the deformation and fracture characteristics of the alloy. To predict behavior under service conditions, a detailed knowledge of the relationships between microstructure and properties is required. Results in this area are limited. For annealed type 304 stainless steel Holmes et al.^{7,8} and Bloom and Stiegler⁹ have related tensile

property changes to microstructural changes; and Bloom and Stiegler¹⁰ have examined the effects of irradiation on creep-rupture properties. The results can be summarized as follows:

1. The changes in tensile strength properties for irradiation and test temperatures below about 600°C can be correlated with the void-dislocation structure.⁷⁻⁹
2. The loss of tensile ductility at temperatures below about 550°C is related to reduced work hardening coefficients. Above 650°C helium embrittlement limits ductility. Between 550 and 650°C both mechanisms may be important.⁹
3. A significant loss of creep-rupture ductility occurs for irradiation and test temperatures in the range 550 to 750°C. At the lower temperatures (below about 650°C) this ductility loss is not attributable to helium alone but is also related to the void-precipitate structure.¹⁰

Few results are available for type 316 stainless steel which is a more complex alloy due to phase instabilities. Barton and Higgins¹¹ have related the increase in tensile yield stress of annealed type 316 stainless steel irradiated in the temperature range 420 to 650°C to the presence of the void-dislocation structure. With regard to creep-rupture properties,¹²⁻¹⁶ the response of the alloy to irradiation is strongly sensitive to both irradiation and test temperature as well as initial microstructure. These investigations have shown that irradiation produces decreased ductility and rupture life. No systematic attempt has been made to relate changes in creep-rupture properties to microstructural changes.

In the present study, annealed and cold-worked type 316 stainless steel was irradiated at temperatures in the range of interest to LMFBR's to a maximum fluence of 2.7×10^{22} neutrons/cm² (> 0.1 MeV). Changes in microstructure that occurred during irradiation were characterized and related to the changes in creep deformation and fracture behavior.

EXPERIMENTAL PROCEDURE

The chemical composition of the type 316 stainless steel used in this investigation is listed in Table 1. The material was received as 1/2-in.-diameter mill-annealed rod. It was swaged (at room temperature) to 0.395 in. diameter, annealed 1 h at 1200°C, and swaged to 0.280 in. diameter (about 50 percent cold work). Test specimens with 0.125 in. diameter by 1-in.-long gage sections were machined from a portion of the rod at this point. These specimens were either left in the 50 percent cold-worked condition or given the desired annealing treatment. The remainder of the 0.280-in.-diameter rod was annealed 1 h at 1050°C and swaged to give material with 20 percent cold work from which test specimens were then machined.

Specimens were irradiated in the Experimental Breeder Reactor-II (EBR-II) to a maximum fluence of 2.7×10^{22} neutrons/cm² (> 0.1 MeV). Irradiation temperatures were in the range 450 to 825°C. 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. (See references 10 and 15 for more complete description of the irradiation experiment). In some cases changes in temperature occurred due to differential swelling

between the containment tube and specimen holder. The temperatures were calculated using both the initial (preirradiation) and final (postirradiation) dimensions of the containment tubes and specimen holders. In those cases where the calculations showed that a change in temperature greater than 10°C had occurred during irradiation, the irradiation temperatures are given as (Initial Temperature)/(Final Temperature) (e.g., 675/715°C). These calculated irradiation temperatures were checked by lattice parameter measurements on silicon carbide samples which were contained in several of the specimen holders. The detailed results are given elsewhere.¹⁵ The accuracy of the calculated temperatures is dependent on the temperature and is estimated to be $\pm 15^\circ\text{C}$ at 450°C, 30°C at 600°C, and $\pm 50^\circ\text{C}$ at 750°C.

The microstructure of the material in the as-irradiated condition was characterized by transmission electron microscopy of samples which were removed from the shoulders of the mechanical property specimens prior to testing. Examination of tested specimens included optical metallography, transmission electron microscopy of samples removed from both the stressed and unstressed portions of the test specimens, and scanning microscopy examination of the fractures.

Quantitative measures of defect concentrations were obtained by determining foil thicknesses by stereomicroscopy. For loop and dislocation counts both micrographs of a stereo pair were made in the same reflection (usually [200]) and an image in a second reflection (usually [020]) was obtained to ensure that all dislocations were being observed. Counts were made on composite micrographs showing

all dislocations or allowance was made for invisible dislocations by assuming all Burgers vectors were equally probable. No evidence was obtained to dispute this.

Postirradiation creep-rupture tests were performed 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. Creep-rupture tests were also performed on unirradiated specimens in the as-heat treated condition.

RESULTS

Microstructures of Irradiated Specimens

Annealed Material

The microstructural changes introduced into annealed type 316 stainless steel were qualitatively similar to those produced in type 304 in that void and dislocation structures developed and precipitation reactions were enhanced and modified. There were, however, important differences between the two steels in numbers and sizes of the various defects. Statistics describing the various defect configurations measured in this study are summarized in Table 2. Microstructures are summarized in Fig. 1.

At 455 and 510°C faulted dislocation loops and voids were found. In some grains enhanced loop formation was noted in regions adjacent to grain boundaries. Occasional blocky $M_{23}C_6$ precipitate particles were found in grain boundaries, but little precipitation was noted within the grains.

For irradiation at 580°C a mixture of faulted and unfaulted dislocation loops and dislocation lines was observed. It was not possible to characterize the distribution of unfaulted loops, and they were included in the count of dislocation lines which yielded a dislocation density of 4×10^9 cm/cm³. Most of the voids found in this specimen were associated with precipitate particles. Not all precipitates had voids attached, however. Two types of precipitate particle were present: a blocky $M_{23}C_6$ carbide and some needle or, more properly, ribbon-like particles. Although selected area diffraction patterns have been obtained from the latter, it has been impossible to match them with any of the known phases in this system; namely, $M_{23}C_6$, M_6C , MC, laves, chi or sigma. It is possible that the precipitate is a variant of one of these phases having a composition and lattice parameter different from that usually found. Alternatively, it may be a new phase not formed by conventional thermal treatments. The ribbon-like precipitates are parallel to [100] directions. It has not been possible to specify their habit plane, but Bisson and Vouillon¹⁶ have found similar precipitates and from a comparison with stacking fault fringes concluded that they lay on {110} planes. In their case these precipitates disappeared on annealing at temperatures above 600°C. Here they were found for irradiations at a calculated temperature of 630°C. The ribbon-like precipitates were present in a concentration of 3.6×10^{13} /cm³. The $M_{23}C_6$ particles were present at a level of 1.3×10^{12} /cm³ and there were about 1×10^{13} voids/cm³. Every carbide had a void attached as did about a quarter of the ribbon-like precipitates. In some cases voids were found at the center of three mutually perpendicular ribbon-like precipitates, and in a

few instances several voids were distributed along a single precipitate. A nearly continuous distribution of $M_{23}C_6$ particles was found along the grain boundaries in this specimen.

The specimen irradiated at 675/715°C contained a cavity structure which had a bimodal size distribution, small cavities ranging up to 300 Å in diameter usually, although not always, associated with plate-like $M_{23}C_6$ particles and large cavities up to 1000 Å in diameter on angular precipitates, probably also carbides. Because of their small size the former are believed to be equilibrium helium bubbles. The latter, which were present in a relatively low concentration, $< 10^{12}/\text{cm}^3$, may be voids. Grain boundaries displayed an almost continuous layer of small $M_{23}C_6$ particles. No dislocation loops were found in this specimen, but a general dislocation density of $9.8 \times 10^8 \text{ cm}/\text{cm}^3$ was measured. In some cases loops were observed that evidently had been punched out from large precipitate particles during cooling, but these were isolated and were not included in the dislocation count.

Cold-Worked Material

The microstructures in specimen which were irradiated in the 20 percent cold-worked condition (Fig. 2) were significantly different from those present in specimen irradiated in the annealed condition. Void formation was suppressed except in small recovered areas of a specimen irradiated at 580°C. The formation of dislocation loops was eliminated under all conditions, and the type, amount,

and distribution of precipitate particles was altered as compared to specimen irradiated in the annealed condition.

It was not possible to detect any microstructural change in a specimen irradiated at 450°C to a fluence of 1.9×10^{22} neutrons/cm². It was somewhat easier to image the microstructure than in the as-cold worked condition, but this cannot be taken as evidence for recovery. No precipitate particles were observed either on grain boundaries or within grains.

For irradiation at 510°C to a fluence of 1.2×10^{22} neutrons/cm² some recovery clearly had taken place in the dislocation structure. In addition, sheets of $M_{23}C_6$ formed on the stacking fault bands, and some elongated laves phase particles were observed on or near the bands. Isolated $M_{23}C_6$ particles were also located along the grain boundaries.

More extensive carbide and laves phase precipitation in the same morphologies was present in the specimen irradiated at 580°C to a fluence of 2.7×10^{22} neutrons/cm². In addition, more extensive recovery had occurred, and in regions of relatively low dislocation density some voids were found. No loops were evident anywhere. There were such extreme variations in local dislocation, void, and precipitate concentrations that no meaningful numbers could be extracted to describe the specimen.

For irradiation to a fluence of 2.3×10^{22} at 680/725°C extensive recovery, some recrystallization and precipitation of large sigma phase particles occurred. No cavities were

found in any of the recrystallized grains, but the amount of recrystallization was significantly less than in unirradiated control specimens held 4000 h at 550°C.

Recrystallization was not complete in a specimen irradiated to a fluence of 2.7×10^{22} at a temperature that ranged between 735°C at the start of the irradiation and 795°C at the finish. Other studies have shown that in unirradiated samples recrystallization is essentially complete after 10 h at 750°C.¹⁷ These results suggest that the initial irradiation temperature was probably less than 735°C. Bubbles were noted in the recrystallized grains and especially in the interface between recovered and recrystallized material. Laves phase particles were also found in both areas; in recovered regions they were elongated as described previously, but in recrystallized areas they were equiaxed.

Effect of Irradiation on Creep-Rupture Properties

The effects of fast neutron irradiation on the rupture life and creep rate of material irradiated in the annealed and 20 percent cold-worked conditions are shown in Figs. 3 and 4. The solid lines represent the properties of the unirradiated material. For irradiation in the annealed condition, the rupture life was decreased by a factor of 3 or less in the temperature range 550 to 700°C. At 750°C the rupture life was reduced by 1 to 2 orders of magnitude with the effect being greater at the higher stress levels. The creep rates and stress dependency of the creep rate were not significantly affected by irradiation in the temperature range 550 to 700°C. The creep rate was, however, markedly increased at 750°C.

When irradiated in the cold-worked condition, the changes in creep-rupture properties were quite complex. The rupture life was increased and creep rates decreased at 550°C, while at 600°C the opposite trend was observed. At 700 and 750°C the limited data suggest a decreased rupture life and increased creep rate at high stress levels with the effect becoming less and possibly reversing at lower stress levels.

Creep strain at the beginning of tertiary creep and total creep elongation for the unirradiated and irradiated conditions are shown in Fig. 5. When irradiated in the annealed condition, the specimens were less ductile at all temperatures in the range 550 to 750°C. For cold-worked material, the ductility after irradiation was reduced at 550 to 650°C but was increased sharply and equaled or exceeded the as-cold worked values at 700 and 750°C. This latter effect appears to be related to recovery and recrystallization during irradiation.

Deformation and Fracture

Optical Metallography

Longitudinal sections of samples irradiated in the annealed condition and tested to failure at 550, 650, and 750°C are shown in Fig. 6. At each temperature extensive cracking occurred on grain boundaries approximately normal to the applied load. Cracks such as those shown in Fig. 6 were not localized to the region of the fracture but were found along the entire gage section. At 550 and 600°C a crack, particularly in a region removed from the fracture,

was not wedge shaped but rather had nearly constant width from one triple point to another. These cracks appeared to have formed and propagated with little sliding on those boundaries inclined to the plane of the crack. Examination of samples in the as-polished and also the etched condition revealed that many of those boundaries normal to the applied load contained isolated cavities. Often a string of cavities could be seen extending from the tip of a grain boundary crack. Although it could not be shown conclusively, it appeared that the cavities formed on or near grain boundary precipitates. A crack then developed as the cavities grew and linked together. At 650°C most of the cracks (including those removed from the fracture) were wedge shaped. Examination of samples in the as-polished condition again revealed small cavities on many of the grain boundaries. At 750°C the cracks were obviously formed by the linking of grain boundary cavities.

Scanning microscopy showed that in the samples irradiated in the annealed condition and tested at 550 and 600°C at least two types of fracture occurred. A portion of the fracture was completely intergranular as shown in Fig. 7(a). Other regions of the fracture consisted of areas of intergranular fracture surrounded by ductile transgranular fracture (Fig. 7(b)). It appears that the failure was initiated by the formation of grain boundary cracks which propagated and eventually reduced the effective cross section of the specimen. The final separation then occurred by the linking of these cracks at a higher strain rate with considerable transgranular fracture.

For samples irradiated in the 20 percent cold-worked condition and then tested in the range 550 to 650°C, the grain boundary cracks were fewer in number than in the companion samples irradiated in the annealed condition [Fig. 8(a)]. The cracks in the cold-worked specimens were generally narrower than in the annealed specimens, and the percentage of intergranular fracture was much larger (Fig. 9). Such observations suggest strongly that the strength of the matrix is important in crack initiation, propagation, or both. At 750°C [Fig. 8(b)] extensive sigma phase precipitation was apparent and cavities were usually associated with the sigma particles. The sigma phase particles were never fractured; the cavities and cracks were located at the particle-matrix interface.

Transmission Microscopy

Dislocation structures present in the gage section of annealed specimens irradiated and tested in the range 550 to 750°C are shown in Fig. 10. In the specimen irradiated at 580°C and then tested at 550°C and 45,000 psi ($t_R = 452$ h, $\epsilon_f = 3.6$ percent) neither the void size nor concentration had changed appreciably during the test, but the dislocation density had increased by about a factor of 10 over the as-irradiated value to 4×10^{10} cm/cm³. No faulted loops were visible in the matrix although some were present prior to testing. Subboundaries, although not well defined, appeared to be forming in many areas. For example, in Fig. 10(a) areas relatively free of dislocations surrounded by heavily tangled regions are apparent. The dislocation substructure did not appear

to be altered near grain boundaries. Very similar dislocation structures were found in a specimen which was tested at 600°C and 40,000 psi ($t_R = 199$ h, $\epsilon = 3.4$ percent). In each case an almost continuous precipitate was present at the grain boundary. This precipitate was present in the as-irradiated condition and was identified as $M_{23}C_6$. The specimen tested at 650°C did not contain a uniform dislocation substructure. In some areas a well defined cell structure was present as shown in Fig. 10(b). Because of thinning difficulties large areas of foil were not examined and an average subgrain diameter was not obtained, but subgrain diameters in the range 0.5 to 5 μm were present. Other areas of the same specimen contained more uniform dislocation distributions. At 750°C isolated dislocations were present in regions removed from precipitates, and dislocation tangles were present around precipitate particles. No cell formation was observed.

DISCUSSION

Neutron irradiation to fluence levels in the range 1.2 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) caused changes in creep rate, rupture life, and ductility, the extent of which were dependent on the irradiation temperature and initial microstructure.

Changes in creep rate were rather small with two exceptions. First, irradiation in the 20 percent cold-worked condition at 510°C followed by testing at 550°C resulted in a decrease in minimum creep rate by about an order of magnitude. Examination of microstructures in the as-irradiated condition indicated that the dislocation density

had decreased slightly during the irradiation. The reasons for the decreased creep rate were not apparent from transmission electron micrographs. Second, irradiation in the annealed condition at 735/795°C followed by testing at 750°C and irradiation in the 20 percent cold-worked condition at 680/725°C and 735/795°C followed by testing at 700 and 750°C resulted in creep rates about an order of magnitude higher than in the respective as-heat treated conditions. For the annealed material, the loss of strength during irradiation resulted from precipitation of carbon as massive $M_{23}C_6$ particles. This prevented carbide precipitation on the strain-induced dislocation structure during the postirradiation test and thus reduced the strength.^{18,19} In the cold-worked condition, the recovery and recrystallization that occurred during irradiation caused the decrease in strength. This is substantiated by the results on 20 percent cold-worked specimens that were aged 4000 h at 650°C. For this aging treatment about 20 percent recrystallization had occurred and in the unrecrystallized areas the dislocation density has decreased noticeably and some subboundaries had formed. The creep rates were increased by about a factor of 6 over the as-cold-worked condition. There are two additional factors that may affect creep strength at the higher irradiation and test temperatures. The removal of molybdenum and chromium from solution by the formation of sigma and laves intermetallic phases would reduce the solid solution strengthening. Also, an *apparent* increase in creep rate results from the reduced ductility and rupture life. For example, the specimens irradiated in the annealed condition and then tested at 750°C and 10,000 psi

failed in 4 h as compared to about 600 h for the unirradiated specimen. If irradiation had no effect on the strain-time behavior then the irradiated specimen failed while still in primary creep (which lasted about 60 h in the unirradiated specimen). Thus, it is probable that a minimum or secondary creep rate characteristic of the irradiated microstructure was not established prior to fracture. This is suggested by the low dislocation density and lack of subboundaries in the irradiated samples that were tested at 750°C. Other investigators²⁰ have shown that in unirradiated samples tested at this temperature well defined subboundaries form shortly after second-stage creep is established.

Ductility as measured either by total elongation or strain at the initiation of tertiary creep was reduced by irradiation for all test conditions. The magnitude of this effect as well as the mechanism responsible for it appeared to vary with temperature.

In annealed specimens irradiated and tested at 550 to 600°C and in 20 percent cold-worked specimens irradiated and tested at 550 to 650°C the fractures were predominantly intergranular. Grain boundary cracks in regions removed from the fracture *were not wedge shaped* but remained very narrow in agreement with the observation that the total amount of deformation was small. Optical and scanning microscopy showed that the cracks were probably formed by the linking of cavities that formed on the grain boundaries. Once formed, extensive propagation of the crack along the grain boundary occurred. The fact that the length of the cracks as well as the percentage of intergranular

fracture was increased in the 20 percent cold-worked condition suggests that an increased matrix strength increases the rate of crack propagation.

The appearance of wedge-type cracks in annealed specimens irradiated and tested at 650°C indicates that matrix deformation and grain boundary sliding were increased as compared to annealed specimens irradiated and tested at 550 and 600°C or cold-worked specimens at 550, 600, and 650°C. The increased matrix deformation appeared to limit the propagation of a crack from one triple grain junction to another and in effect made fracture more difficult than for lower irradiation and test temperatures. As a consequence, the ductility increased in the temperature range 650 to 700°C. At the highest test temperature, 750°C, metallographic examination showed that fracture was also caused by the linking of cavities on boundaries normal to the applied load. The embrittlement appeared to result from the stress induced growth of grain boundary helium bubbles. The higher ductility for specimens irradiated in the cold-worked condition at 700 and 750°C as compared to the annealed condition may be due to the fine grain size developed in this material as a result of extensive recovery and recrystallization during irradiation.²¹

From these observations and from the published work of other investigators it is possible to develop the rudiments of a model for the combined effects of initial microstructure, irradiation temperature, neutron fluence, and transmutation-produced helium upon the intergranular fracture mechanism.

At all irradiation and test temperatures the grain boundary cracks appear to be initiated by the linking of cavities that form along the boundary. The nuclei for the cavities were not positively identified in the present study. However, based on the present and previous results it is reasonable to assume that helium was present at the grain boundaries of the irradiated samples.^{2,3} For those alloys and irradiation conditions which form a grain boundary precipitate such as the $M_{23}C_6$ carbide in this study, the helium will often precipitate as bubbles at the precipitate-matrix interfaces.²² When no precipitates are present the helium still precipitates as bubbles in the boundary, particularly at triple grain junctions. Stress-induced growth of a bubble to form a cavity can occur by vacancy condensation. Speight and Harris²³ have developed an expression for the rate of cavity growth (no internal pressure) on boundaries across which a normal stress acts. This expression can be modified to account for the internal pressure present in a helium bubble. The result is

$$\frac{dR_o}{dt} \approx \frac{zD_g\Omega}{2kTR_o} \frac{(\sigma + P - \frac{2\gamma}{R_o})}{\left[\ln\left(\frac{\eta}{2R_o}\right) - \frac{\eta^2 - 4R_o^2}{2\eta^2} \right]} \left[\frac{1}{R_o} - \frac{4R_o}{\eta^2} \right] \dots (1)$$

where

R_o = radius of the cavity,

t = time,

z = grain boundary thickness,

D_g = grain boundary diffusion coefficient,

Ω = atomic volume,

k = Boltzmann's constant,

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

σ = stress normal to the grain boundary,

P = gas pressure in the cavity = $2\gamma R_1^2/R_0^3$, where R_1 is the original radius of the equilibrium gas bubble,

γ = surface tension, and

η = the bubble spacing.

Taking values of $T = 823^{\circ}\text{K}$, $zD_g = 5 \times 10^{-18} \text{ cm}^2/\text{sec}$,²⁴ $\sigma = 3 \times 10^9 \text{ dyne/cm}^2$ (45,000 psi), $\gamma = 1500 \text{ dyne/cm}$, and an estimated initial equilibrium gas bubble radius of $3.5 \times 10^{-7} \text{ cm}$ and spacing of 10^{-4} cm (estimated from micrographs), one obtains a cavity growth rate (dR_0/dt) of $1.5 \times 10^{-8} \text{ cm/sec}$ at a cavity radius of $100 \text{ \AA}$. Growth rates thus appear to be sufficiently large to account for the cavities observed on grain boundaries. The eventual linking of the cavities to produce a crack could thus occur with no significant grain boundary sliding or matrix deformation. As pointed out previously, crack propagation appears to be dependent on the strength of the matrix. A possible reason for this behavior is that the stress concentration near the crack tip in strong specimens could cause an increase in growth rate for cavities in this region. The stress distribution ahead of a crack in a plastically deforming material has not been determined analytically. Stress concentrations do exist however, and motion

of dislocations near the crack tip can cause blunting and relaxation of the local stress.^{25,26} For specimens irradiated at conditions which produced relatively high flow stresses, this relaxation could be inhibited and the stress concentration could thus assist crack propagation by the increased growth rate of cavities near the crack tip. Irradiation and test conditions that result in lower flow stresses would allow relaxation of the stress and thereby decrease the rate of crack propagation. For the highest irradiation and test temperatures, the stress induced growth of bubbles to form cracks occurs very rapidly, and rupture lives are reduced by about 2 orders of magnitude.

The shape of a crack as observed in a fractured specimen is dependent upon the matrix and grain boundary deformation which occurs subsequent to crack formation but before the final fracture. At the low temperatures the strength of the irradiated specimens limits deformation and the cracks remain very narrow. At 650°C the strength of material irradiated in the annealed condition is lower, and for these conditions grain boundary sliding and matrix deformation assist in opening the cracks to give them a wedge shape. At the highest temperature fracture occurs by the linking of cavities before there is time for significant creep deformation to occur, as confirmed by the very low dislocation densities.

CONCLUSIONS

Fast neutron irradiation of type 316 stainless steel at temperatures in the range 450 to 850°C to peak fluences of 2.7×10^{22} neutrons/cm² (> 0.1 MeV) produced major changes in microstructure and creep-rupture properties. The extent of these changes was strongly dependent upon the temperature and the preirradiation microstructure. Annealed materials irradiated below about 650°C contained a structure of voids and dislocations. In addition, $M_{23}C_6$ carbides formed on grain boundaries and in the matrix, and a ribbon-like precipitate that could not be identified occurred often in association with the voids. The presence of the latter precipitate again illustrates⁵ that the nature of the precipitates that form or the kinetics of the reactions or both are altered during neutron irradiation. Above 650°C large $M_{23}C_6$ carbides, sigma phase particles, and helium bubbles were the main microstructural features in specimens irradiated in the annealed condition. In the 20 percent cold-worked condition voids were observed only at an irradiation temperature of 580°C, and these voids were generally located in recovered regions having relatively low dislocation densities. Precipitation was markedly altered in type, amount, and distribution by cold working.

The most important effect of irradiation on the creep-rupture properties was a decrease in ductility, both the strain at initiation of tertiary creep and the total elongation, particularly at temperatures in the range 550 to 650°C. The low ductilities were accompanied by pronounced intergranular fracture. At all temperatures the formation of grain boundary cracks occurred by the stressed-induced growth

and linking of grain boundary cavities. Helium bubbles are believed to serve as cavity nuclei, and stress-induced growth to occur by the vacancy-diffusion mechanism proposed by Speight and Harris.²³ Propagation of these cracks was dependent upon the flow stress of the matrix. At the lower irradiation temperatures, which resulted in high flow stresses, cracks were narrow and extended large distances (often across several triple grain junctions), possibly as a result of increased cavity growth rates near the leading edge of a crack due to stress concentration at the crack tip. At slightly higher temperatures this stress concentration was apparently reduced by plastic deformation, and the crack propagation was sufficiently reduced to allow grain boundary sliding and matrix deformation to occur. At the highest temperatures cavity growth rates were sufficiently rapid to reduce the rupture life by about a factor of 100.

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FIGURE CAPTIONS

- Fig. 1. Microstructure of Type 316 Stainless Steel Irradiated in the Annealed Condition to Fast Neutron Fluences in the Range 1.5 to 1.9×10^{22} neutrons/cm² (> 0.1 MeV).
- Fig. 2. Microstructure of Type 316 Stainless Steel Irradiated in the 20 Percent Cold-Worked Condition to Fast Neutron Fluences in the Range 1.2 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV).
- Fig. 3. Effect of Irradiation to Fast Neutron Fluences in the Range 1.5 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) on the Creep-Rupture Properties of Annealed Type 316 Stainless Steel. Solid lines represent unirradiated properties. Specimens were irradiated within $\pm 30^\circ\text{C}$ of respective test temperatures.
- Fig. 4. Effect of Irradiation to Fast Neutron Fluences in the Range 1.5 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) on the Creep-Rupture Properties of 20 Percent Cold-Worked Type 316 Stainless Steel. Solid lines represent unirradiated properties. Specimens were irradiated within $\pm 30^\circ\text{C}$ of respective test temperatures.
- Fig. 5. Effect of Irradiation on the Creep-Rupture Ductility of Type 316 Stainless Steel.
- Fig. 6. Creep-Rupture Fractures of Type 316 Stainless Steel Irradiated in the Annealed Condition. (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 , 7500 psi.

Fig. 7. Scanning Electron Micrographs of Fracture in Type 316 Stainless Steel Specimen Irradiated at 580°C to 1.9×10^{22} neutrons/cm² and Tested at 550°C and 45,000 psi. (a) Area of intergranular fracture. (b) Area of inter- and transgranular fracture.

Fig. 8. Creep-Rupture Fractures in Type 316 Stainless Steel Irradiated in the 20 Percent Cold-Worked Condition. (a) Irradiated at 580°C; tested at 600°C and 40,000 psi stress. Etching reveals extensive carbide precipitation on deformation bands. (b) Irradiated at 735/795°C; tested at 750°C and 7500 psi stress. Precipitates visible in etched condition are sigma phase.

Fig. 9. Intergranular Fracture of Type 316 Stainless Steel Irradiated in the 20 Percent Cold-Worked Condition at 580°C and Tested at 600°C and 40,000 psi.

Fig. 10. Dislocation Structures in Annealed Type 316 Stainless Steel Irradiated and the Creep-Rupture Tested in the Range 550 to 750°C. (a) Tested at 550°C; 45,000 psi. (b) Tested at 650°C; 25,000 psi. (c) Tested at 750°C; 7500 psi.

Fig. 1. Microstructure of Type 316 Stainless Steel Irradiated in the Annealed Condition to Fast Neutron Fluences in the Range 1.5 to 1.9×10^{22} neutrons/cm² (> 0.1 MeV).

450°C
1.9 × 10²²

510°C
1.2 × 10²²

580°C
2.7 × 10²²

725°C
2.3 × 10²²

Fig. 2. Microstructure of Type 316 Stainless Steel Irradiation in the 20 Percent Cold-Worked Condition to Fast Neutron Fluences in the Range 1.2 to 2.7 × 10²² neutrons/cm² (> 0.1 MeV).

Fig. 3. Effect of Irradiation to Fast Neutron Fluences in the Range 1.5 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) on the Creep-Rupture Properties of Annealed Type 316 Stainless Steel. Solid lines represent unirradiated properties. Specimens were irradiated within $\pm 30^\circ\text{C}$ of respective test temperatures.

Fig. 4. Effect of Irradiation to Fast Neutron Fluences in the Range 1.5 to 2.7×10^{22} neutrons/cm² (> 0.1 MeV) on the Creep-Rupture Properties of 20 Percent Cold-Worked Type 316 Stainless Steel. Solid lines represent unirradiated properties. Specimens were irradiated within $\pm 30^\circ\text{C}$ of respective test temperatures.

Fig. 5. Effect of Irradiation on the Creep-Rupture Ductility of Type 316 Stainless Steel.

(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, 7500 psi.

Fig. 6. Creep-Rupture Fractures of Type 316 Stainless Steel Irradiated in the Annealed Condition.

Fig. 7. Scanning Electron Micrographs of Fracture in Type 316 Stainless Steel Specimen Irradiated at 580°C to 1.9×10^{22} neutrons/cm² and Tested at 550°C and 45,000 psi. (a) Area of intergranular fracture. (b) Area of inter- and transgranular fracture.

(a) Irradiated at 580°C; tested at 600°C and 40,000 psi stress. Etching reveals extensive carbide precipitation on deformation bands.

(b) Irradiated at 735/795°C; tested at 750°C and 7500 psi stress. Precipitates visible in etched condition are sigma phase.

Fig. 8. Creep-Rupture Fractures in Type 316 Stainless Steel Irradiated in the 20 Percent Cold-Worked Condition.

Fig. 9. Intergranular Fracture of Type 316 Stainless Steel
Irradiated in the 20 Percent Cold-Worked Condition at 580°C and
Tested at 600°C and 40,000 psi.

(a) Tested at 550°C; 45,000 psi.

(b) Tested at 650°C; 25,000 psi.

(c) Tested at 750°C; 7500 psi.

Fig. 10. Dislocation Structures in Annealed Type 316 Stainless Steel Irradiated and the Creep-Rupture Tested in the Range 550 to 750°C.

Table 1—*Chemical Composition of Type 316 Stainless Steel*

Element	Wt %
Cr	18.0
Ni	13.0
Mo	2.58
Mn	1.9
Si	0.8
Ti	0.05
C	0.05
P	0.013
S	0.016
N	0.05
B	0.0005

Table 2—Defect Statistics in Annealed Type 316 Stainless Steel after Irradiation

Irradiation Temperature (°C)	Fluence Neutrons/cm ² (> 0.1 MeV)	Dislocation Structures			Voids		$\frac{\Delta V}{V}$ Calc (%)	Precipitates	
		Concentration	Size	Dislocation Density (cm/cm ³)	Concentration (voids/cm ³)	Mean Diameter (Å)		Type	Concentration
455	1.9×10^{22}	$1 \times 10^{15}/\text{cm}^3$	< 100 Å min 1700 Å max	—	6.9×10^{13}	236	0.06	—	—
510	1.5×10^{22}	$5.2 \times 10^{14}/\text{cm}^3$	125 Å min 2100 Å max	—	1.8×10^{13}	337	0.05	—	—
580	1.9×10^{22}	$4.8 \times 10^{12}/\text{cm}^3$	870 min 1700 ave 3500 max	4×10^9	1×10^{13}	666	0.2	M ₂₃ C ₆ Ribbon-like	1.3×10^{12} 3.6×10^{13}
675/715	1.9×10^{22}	—	—	9.8×10^8	7.7×10^{12a}	—	—	M ₂₃ C ₆ Rod-like	1.8×10^{12} 1.6×10^{11}
735/795	2.7×10^{22}	—	—	—	—	—	—		b

^aHelium bubbles.

^bSample was not examined by transmission microscopy. Optical metallography revealed extensive precipitation both at grain boundaries and within matrix.