FATIGUE BEHAVIOR AT 650°C OF 20%-COLD-WORKED TYPE 316 STAINLESS STEEL IRRADIATED AT 550°C IN THE HFIR*

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ABSTRACT

Type 316 stainless steel in the 20%-cold-worked condition was irradiated in the High Flux Isotope Reactor (HFIR) and subsequently tested in fatigue. The specimens were irradiated at 550°C to damage levels of 8 to 12 dpa and transmutation helium levels of 300 to 500 at. ppm. Fatigue testing at 650°C revealed that cyclic life was not significantly affected by the irradiation. However, unlike the results of tests of the same material at 550°C, no endurance limit was observed. The absence of an endurance limit is interpreted in terms of thermal creep.

KEY WORDS: Fatigue, Irradiation, Fusion, Helium, Stainless Steel, Radiation Damage

INTRODUCTION

Fatigue is a concern in any fusion device that operates in a cyclic mode. Moreover, even in a steady state device, startup and shutdown cycles as well as plasma disruptions will introduce high thermal stress transients. As a result of such transients, low-cycle fatigue properties are a concern as well as high-cycle fatigue resulting from normal cyclic operation.

In addition to the stress cycles applied to first wall and blanket structures, a unique and severe radiation environment must be endured. The intense flux of neutrons, with energies at high as 14.1 MeV, causes atomic displacements as well as transmutation reactions. High energy neutron displacement damage has been studied at some length and has been found to result in a multiple cascade microstructure with subcascades similar to cascades resulting from neutrons in fission reactors.¹ The differences are understood and can be accounted for. Many metals used in structural alloys undergo (n,α) and (n,p) reactions at neutron energies above about 10 MeV. Hydrogen resulting from (n,p) reactions diffuses rapidly at elevated temperatures, and is not perceived to be a problem in austenitic steels at present; generally austenitic stainless steels are very resistant to hydrogen embrittlement. Helium, however, is insoluble in metals and is known to segregate to sinks such as grain boundaries. For this reason, the effects of helium on candidate fusion materials must be investigated.
The present study investigates fatigue in 20%-cold-worked AISI type 316 stainless steel (20%-CW 316 SS) that has been irradiated in the High Flux Isotope Reactor (HFIR). The high thermal flux in this reactor produces helium through a two-step transmutation reaction beginning with $^{58}$Ni. The accompanying fast flux simultaneously produces atomic displacement damage.

Previous studies have investigated fatigue in the same material irradiated at 430°C (ref. 2) and 550°C (ref. 3). Irradiation in the HFIR was found to reduce fatigue life by a factor of 3 to 10 at 430°C and to have no significant effect at 550°C. However, in these investigations, unlike the present study, the material was tested at the irradiation temperature. In the case of accidental thermal transients such as plasma disruptions or local hot spots that develop from temporary coolant flow restriction, structural materials even in the blanket could experience temperature excursions. Projections of the frequency of plasma disruptions vary widely, but the thermal stresses produced may be well into the low-cycle fatigue regime. Since irradiated materials stressed above their irradiation temperature often experience severe degradation in mechanical properties, fatigue properties at a temperature 100°C higher than the irradiation temperature were investigated.

**EXPERIMENTAL PROCEDURE**

**Specimen Preparation**

Specimens were prepared from a reference heat (X15893) of AISI type 316 stainless steel being used in the U.S. Fusion Materials Program. The composition is given in Table I. Following a vacuum anneal at 1050°C
for 1 h the material was cold swaged to 20% reduction in area. This
treatment resulted in an average grain size of 40 μm. The resulting
6.43 mm rods were machined into specimens with an hourglass gage section
3.18 mm in diameter. The specimen geometry is shown in Fig. 1.

Irradiation

In the HFIR the fast portion of the neutron spectrum produces
atomic displacement damage, and the thermal portion results in helium
production through the following reactions:

\[ ^{58}\text{Ni} (n_{th},\gamma) ^{59}\text{Ni} , \]
\[ ^{59}\text{Ni} (n_{th},\alpha) ^{56}\text{Fe} . \]

The helium is deposited homogeneously and simultaneously with displace-
ment damage. The specimens were irradiated in a position where both
thermal and fast (E > 0.1 MeV) fluxes exceed \(10^{19} \text{n/(m}^2\cdot\text{s})\).

The specimens were arranged ten per irradiation capsule along the
longitudinal axis. Figure 2 shows the positioning of a specimen
surrounded by a helium gas gap to control radial heat conduction, thus
providing the desired elevated temperature through nuclear heating. Low
melting metals and alloys and silicon carbide were used as temperature
indicators as described in previous reports.\(^1,5\) The melt wire materials
used were Cu-30.7 Mg, Mg-23.5 Ni, Al-33 Cu, Al-11.7 Si, and Zn. The
irradiation temperature was determined to be 550 ± 25°C.

Irradiation and test parameters are given in Table II. Damage
levels were calculated using the method recommended by the IAEA working
group\(^6\) based on previous dosimetry measurements, and helium levels were
calculated from a relation based on experimental data.\(^7\) An increment
in dpa produced by the recoiling iron nucleus in the $^{58}$Ni (n$_{th}$,a) $^{56}$Fe reaction was added using the relation derived by L. R. Greenwood.\(^8\)

**Fatigue Testing**

The tests were performed on a servo-hydraulic closed-loop controlled testing system installed in a radiation hot cell. The system has a four-column load frame capable of 220 kN (50,000 lb). It is equipped with an ultrahigh vacuum system pumped by a turbomolecular pump capable of pressures below $10^{-6}$ to $10^{-4}$ Pa during elevated-temperature testing. Specimen heating is accomplished by RF-induction with a load coil surrounding the specimen. Strain is measured by a diametral extensometer which fits between two windings of the load coil.

Tests were performed at 650°C, which is 100°C above the irradiation temperature. Specimens were subjected to a triangular strain versus time program beginning with compression at a strain rate of $4 \times 10^{-3}$ s$^{-1}$. For low-cycle fatigue ($\Delta \varepsilon > 0.5\%$), tests were controlled on the basis of axial strain calculated from diametral strain, measured directly at the minimum gage section, through a strain computer. For high-cycle tests, the same strain control was used until a stable hysteresis loop was achieved, at which time control was switched to load. At the same time the frequency was increased by a factor of 10 to reduce the test duration. Specimens were cycled to complete separation in order to perform fractography, except in cases where an apparent endurance limit was observed. All specimens, both irradiated and unirradiated, were loaded remotely using the same procedure in order to avoid differences in alignment.
RESULTS

The test parameters and results are given in Table II. Specimens tested in the low-cycle regime exhibit initial hardening followed by softening during the test. Specimens in high-cycle tests exhibit a nearly constant stress level prior to crack initiation.

Cyclic hardening curves appear in Fig. 3 for the irradiated and unirradiated specimens. The data for the irradiated specimens appear to fall on a single curve irrespective of fluence within the range investigated. The curves for the irradiated and unirradiated specimens are very similar. This is in sharp contrast to cyclic stress-strain curves at lower temperatures from previous work where very significant differences were observed.\(^2,3\) At 430°C the irradiated specimens hardened more than the unirradiated, but at 550°C the irradiated specimens were softer (Fig. 4). Cyclic stress values at 650°C are also nearly the same as those for material irradiated and tested at 550°C.

The cyclic life is presented in Fig. 5. The curves representing the irradiated and unirradiated specimen data are very similar, with the unirradiated curve being slightly above that for the irradiated specimens. Using a least squares fit to a function of the form \( \Delta \epsilon_T = A N_f^{-0.12} + B N_f^{-b} \), the following equations were arrived at:

**Unirradiated:**

\[ \Delta \epsilon_T = 0.019 \times N_f^{-0.12} + 0.92 \times N_f^{-0.60} \]  \hspace{1cm} (1)

**Irradiated:**

\[ \Delta \epsilon_T = 0.016 \times N_f^{-0.12} + 0.29 \times N_f^{-0.46} \]  \hspace{1cm} (2)
where

\[ \Delta e_T = \text{total strain range}, \]
\[ N_F = \text{cycles to failure}. \]

Unlike the material irradiated and tested at 550°C (designated 550/550°C), the material irradiated at 550°C and tested at 650°C (550/650°C) exhibited no endurance limit (10^7 cycle) for strain ranges as low as 0.25%. This is perhaps the most significant difference between the 550/550°C and the 550/650°C test results.

The fracture surfaces of the specimens were examined by scanning electron microscopy (SEM). The most evident fracture surface morphology for both irradiated and unirradiated specimens was transgranular with a tendency toward ductile rupture at high strain ranges. There was also some secondary cracking (Fig. 6), perhaps intergranular, especially in the irradiated specimens. The unirradiated specimen tested at a strain range of 0.25% exhibited cavities which appear to have formed along slip bands (Fig. 7).

**DISCUSSION**

The cyclic life of 20%-CW 316 SS in the 550/650°C tests was very similar to the 550/550°C tests for both the irradiated and unirradiated material. In fact, for design purposes, one curve could be used for both irradiated and unirradiated material for 550 and 650°C. The one significant difference, however, was the apparent absence of an endurance (10^7 cycle) limit at 650°C. In tests performed at 430 and 550°C in a vacuum an apparent endurance limit was observed. Some insight into why an endurance limit failed to appear can be gained from
an interesting microstructural feature which appeared on the fracture surface of the unirradiated specimen tested at the lowest strain range, 0.25%. As shown in Fig. 7, cavities formed on what appear to be slip bands. Since this is an unirradiated specimen tested in the creep regime, the cavities are most likely to be creep voids produced during testing. Such cavities have not been observed in 20%-CW 316 SS either prior to testing or in specimens tested at lower temperatures.

Creep cavities usually nucleate on grain boundaries especially at precipitate interfaces. However, the environment has an effect on their nucleation and growth. Since diffusion of chemically active gases such as oxygen into the specimen accelerates the cavity nucleation or growth rates in creep deformation, cavitation behavior in air is enhanced over that in a vacuum in austenitic stainless steels. The slower nucleation rate of grain boundary cavities could allow sufficient time for persistent slip bands to develop into nucleation sites equally as strong as grain boundaries in trapping vacancies and gas atoms. It would then be possible to form creep cavities and crack initiation sites within the grains.

Because there is strong evidence of creep damage in the material, an effort was made to interpret the absence of an endurance limit in terms of thermal creep. Although no creep rupture data are available on the fusion reference heat of 316 SS, there are data available on two other heats of 316 SS: N-lot and FFTF first core material. Beginning with a general plastic flow law, Johnson et al. have expressed creep rupture life in the following form:

$$\ln t_r = \frac{Q}{RT} + A + \frac{1}{\lambda} \ln \frac{\ln \sigma_s}{\sigma}$$

(3)
where

\[ t_r = \text{rupture life}, \]
\[ T = \text{absolute temperature}, \]
\[ \sigma = \text{hoop stress for tube specimens}, \]
\[ R = \text{universal gas constant}. \]

The material constants have the following values:

\[
\begin{align*}
20\% \text{ N-lot 316 SS}^{12} & \quad 20\% \text{ CW FFTF first core}^{13} \\
Q &= 3.496 \times 10^5 \text{ J/mole} & Q &= 2.688 \times 10^5 \text{ J/mole} \\
A &= -42.94 & A &= -32.69 \\
1/\lambda &= 9.5325 & 1/\lambda &= 7.6812 \\
\sigma^* &= 931 \text{ MPa} & \sigma^* &= 931 \text{ MPa}
\end{align*}
\]

At the maximum stress applied during the fatigue cycle, the above relations predict a creep rupture life at 650°C of the same order of magnitude as the test life. Of course, the specimen does not experience the maximum stress throughout its life, and half the time is spent in compression. An average stress can be chosen to predict life for the tension part of the cycle, but the progression of creep damage in compression is more complicated. Not only is tensile stress not present to assist void growth in compression, but the effect of residual stresses during the following tension cycle resulting from the compression cycle is unknown. Nonetheless, data compiled by Diercks allow a comparison of zero, tension, and compression hold time.\(^{14}\)

These data for 316 SS show little effect of compression hold times as long as 6 min at 650°C. However, similar tension hold times reduce fatigue life by about a factor of 5. Based on this evidence, to simplify the analysis, only the tension portion of the cycle will be assumed to cause creep damage.
The amount of creep damage can then be evaluated using the linear damage concept by the following:

$$\int_0^t \frac{dt}{t_r} = \text{fraction of rupture life expended at time, } t \quad (4)$$

where

$$t_r = \text{rupture life.}$$

It then follows that

$$\int_0^t \frac{dt}{t_r} = 1 \text{ at failure.} \quad (5)$$

Then substituting Eq. (3) into (4) and assuming linear behavior since the strain was in the elastic range,

$$\int_0^t \frac{dt}{t_r} = e^{-(Q/RT + A)} \int_0^t \frac{dt}{(\ln \frac{\sigma^*}{\sigma})^{1/\lambda}} \quad (6)$$

$$= e^{-(Q/RT + A)} \frac{\sigma^*}{\sigma_{\text{max}}} \int_{x_1}^{x_2} \frac{dx}{x^2 (\ln x)^{1/\lambda}} \quad (7)$$

where

$$\tau = \text{period of the ramp fatigue cycle,}$$

$$x = \frac{\sigma^*}{\sigma}$$

Then, expressing the integral in terms of an infinite series we have,

$$\int_{x_1}^{x_2} \frac{dx}{x^2 (\ln x)^q} = \sum_{n=1}^{\infty} \frac{(-1)^n}{(q-1)(q-2)\ldots(q-n)} \frac{x^{q-n}}{(\ln x)^{q-n}} \bigg|_{x_1}^{x_2} \quad (8)$$
Substituting for the constants and computing the first sixteen terms of the infinite series for the case of the 0.25% strain range specimens, the following results are obtained:

\[
\int_{0}^{t} \frac{dt}{t_\tau} = 0.055 \quad -- \text{N-lot} \tag{9}
\]

\[
\int_{0}^{t} \frac{dt}{t_\tau} = 0.35 \quad -- \text{first core.} \tag{10}
\]

The expressions for creep rupture are sensitive functions of materials properties and stress, so that even if creep rupture data were available on the same heat of material such a calculation could only be considered semi-quantitative. Since the tensile properties of the fusion reference heat are closer to those of FFTF first core steel and since the values for creep damage are on the order of several percent to several tens of percent, the amount of creep damage is significant. This result and the observation of creep cavities in the specimen lead to the conclusion that there is significant creep damage in the specimen, and that creep is most likely to be responsible for the absence of an endurance limit at 650°C.

It must be kept in mind, however, that creep damage and fatigue damage are not mutually exclusive. Creep cavities provide stress concentrations that can initiate fatigue cracks, and persistent slip bands (perhaps with precipitates) provide nucleation sites for creep cavities. The preceding analysis was only to demonstrate the significance of creep.

The presence of significant creep damage in the high-cycle fatigue regime indicates the importance of hold period fatigue loading. Helium embrittlement is exacerbated by tension hold periods. Both helium and
creep effects make it important to investigate creep/fatigue loading in
determining the range of applicability of a candidate fusion reactor struc-
tural material at high temperatures.

CONCLUSIONS

1. Irradiation of 20%-CW 316 SS in the HFIR to 12 dpa and 500 at. ppm
He at 550°C does not significantly degrade fatigue life at 650°C.

2. No endurance limit was observed in fatigue testing at 650°C for
irradiated or unirradiated material.

3. Creep damage appears to be an important mechanism of damage at
650°C and responsible for the absence of an endurance limit in contrast
to previous vacuum fatigue tests where an endurance limit was observed.

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REFERENCES


Table I. Composition of AISI Type 316 Stainless Steel Used for the Fatigue Study

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<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
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<tr>
<td>Cr</td>
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<tr>
<td>Mn</td>
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<td>Ni</td>
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<td>Mo</td>
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<td>Co</td>
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<tr>
<td>Cu</td>
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<td>Si</td>
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<table>
<thead>
<tr>
<th>Element</th>
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<td>Nb</td>
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Table II. Irradiation and Fatigue Test Parameters for 20%-Cold-Worked Type 316 Stainless Steel Irradiated in the HFIR and Tested at 650°C

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Fluence (E &gt; 0.1 MeV) (neutrons/m²)</th>
<th>dpa</th>
<th>Helium (at. ppm)</th>
<th>Total Strain Range (%)</th>
<th>Maximum Stress Range at N_F/2 (MPa)</th>
<th>Cycles to Failure</th>
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<tr>
<td>G48</td>
<td>0</td>
<td>2.0</td>
<td>830</td>
<td>1,462</td>
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<td>G47</td>
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<td>AA2C</td>
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<td>800</td>
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<td>G37</td>
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<td>0.70</td>
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<td>AA21</td>
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<td>A79</td>
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<tr>
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<td>400</td>
<td>0.30</td>
<td>440</td>
<td>2,500,000</td>
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Fig. 1. Miniature fatigue specimen. Dimensions are in mm except for angles in degrees and threads in inches.

Fig. 2. Fatigue specimen positioned in irradiation capsule.

Fig. 3. Cyclic stress-strain curves for irradiated and unirradiated 20%-cold-worked type 316 stainless steel. Each point represents an individual specimen.

Fig. 4. Cyclic stress-strain curves for 20%-cold-worked type 316 stainless steel. Specimens were irradiated and tested at 430 and 550°C. The curve for 650°C, irradiated, represents material irradiated at 550°C and tested at 650°C.

Fig. 5. Fatigue life of 20%-cold-worked type 316 stainless steel. Data for both irradiated and unirradiated specimens are shown. Irradiation was done in the HFIR at 550°C, but fatigue testing was done at 650°C.

Fig. 6. Fracture surface of 20%-cold-worked type 316 stainless steel irradiated in the HFIR at 550°C to 8.2 dpa containing 310 at. ppm He. The specimen was tested in fatigue at 650°C ($\Delta \varepsilon_T = 2.0\%$).

Fig. 7. Fracture surface of unirradiated 20%-cold-worked type 316 stainless steel tested in fatigue at 650°C ($\Delta \varepsilon_T = 0.25\%$) showing creep voids along slip bands.
20% COLD WORKED
316 STAINLESS STEEL

\[ T_{irrad} = 550 \, ^\circ C \]
\[ T_{test} = 650 \, ^\circ C \]

- O: \( \phi t = 0 \)
- \( \phi t = 0.85 - 1.4 \times 10^{26} \, n/m^2 \)
**Fig. 4**

**IRRADIATED**

- 430°C
- 550°C
- 650°C

**UNIRRADIATED**

**Cyclic Stress Range, \( \Delta \sigma \) (MPa)**

**Total Strain Range, \( \Delta \varepsilon_t \) (%)**
TOTAL STRAIN RANGE, $\Delta \varepsilon$ (%)

Cycles to Failure

TRIP = 550°C; TEST = 650°C
7 - 44 dpa: 300 - 500 appm HE
$\phi = 0.85 - 1.4 \times 10^{26} \text{ n/mt}^2$
TYPE 316 SS, 20% C.W.
FATIGUE OF HIR IRRADIATED