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The effect of mercury on the fatigue behavior of 316 LN stainless steel

J.P. Strizak [a,*], J.R. DiStefano [a], P.K. Liaw [b], H. Tian [b]

[a] Oak Ridge National Laboratory, Carbon and Insulation Materials Technology Group, Bethel Valley Road,
P.O. Box 2008, Oak Ridge, TN 37831-6088, USA
[b] The University of Tennessee, Knoxville, TN 37996, USA

Abstract

Design of the mercury target system components for the Spallation Neutron Source (SNS) requires data on high- and low-cycle fatigue behavior, and the program in progress includes determining the effects of mercury on the fatigue behavior of type 316 LN stainless steel, the primary material of choice for the target vessel. Uniaxial, load-controlled fatigue tests with $R = -1$ and (minimum stress/maximum stress) have been conducted in air and mercury at room temperature employing constant amplitude, sinusoidal loading at frequencies from 0.1 to 700 Hz. Stress amplitude versus fatigue life (S–N curves) data at 10 Hz for both air and mercury show a sharp knee at approximately 1 million cycles indicating a fatigue endurance limit in either air or mercury around 240 Mpa. Mean stress ($R = 0.1$) lowers the endurance limit to 160 MPa. At relatively low frequency, both frequency and environment (mercury) had some impact on fatigue life of type 316 LN stainless steel at high-stress levels (i.e., stresses considerably above the apparent fatigue limit). Although testing at a high frequency of 700 Hz, showed a decrease in fatigue life in air compared with that at 10 Hz, a significant increase in specimen temperature was observed in air due to self-heating. No pronounced effects of waveform have yet been found, but data are limited. © 2001 Elsevier Science B.V. All rights reserved.

1. Introduction

The target material for the Spallation Neutron Source (SNS) that is under construction at the Oak Ridge National Laboratory in the United States will be liquid mercury that is contained within a type 316 LN SS vessel. The mercury target container will be subjected to a variety of loading conditions in this application and structural design criteria for the target system have been developed which require a consideration of cyclic stresses during service [1]. In support of the data needs, an experimental program is underway to determine the fatigue properties of type 316 LN SS in both air and mercury [2] and for both high- and low-cycle regimes. Previously, screening (fully reversed, load control fatigue) tests conducted in air and mercury at room temperature using a sinusoidal waveform indicated a reduction (2–3 times) in fatigue life in mercury at high-stress amplitudes, but no significant effect on high-cycle fatigue strength (low-stress levels) was observed. Furthermore, examination of the fractures in mercury revealed secondary cracking and little evidence of ductility. If mercury wets the freshly cracked surfaces during fatigue testing and crack growth rate is accelerated (a form of liquid metal embrittlement), such effects could be exacerbated by some experimental parameters.

Generally, when investigating environmental effects, stress amplitude is not the only factor effecting material/environment interaction. Time-dependent environmental effects can also be of importance. When failure occurs by environmental fatigue, stress-cycle frequency, stress-waveshape, and mean stress can synergistically affect the cracking process. The frequency dependence of environmental fatigue is generally thought to result from the fact that interaction of a material and its environment is essentially a rate-controlled process. Low frequencies, when there is substantial elapsed time between

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[*] Corresponding author. Tel.: +1-865 574 5117; fax: +1-865 576 8424.
E-mail address: strizakjp@ornl.gov (J.P. Strizak).

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changes in stress levels, allow time for interaction between material and environment; high frequencies do not. At very high frequencies, self-heating can occur that may affect fatigue behavior. Such effects are normally not considered to be related to an environmental fatigue phenomenon, and experimental design can reduce the temperature in order to qualify the environmental effect at very high frequencies. When environments have a deleterious affect on fatigue behavior, a critical range of frequencies of loading may exist in which mechanical/environmental interaction is significant. Above this range the effect disappears, while below this range the effect may diminish.

S–N (stress-cycle life) fatigue data have been developed for 316 LN stainless steel at room temperature in air and mercury environments over a frequency range 0.1–700 Hz employing a sinusoidal waveform. In addition, limited data were obtained for other waveshapes including a triangular waveform (equal loading and unloading ramp rates) and a positive sawtooth waveform which consists of a slow loading rate to maximum stress followed by fast unloading to complete the stress-cycle.

2. Experimental

2.1. Material, specimens, and testing equipment

A single heat of mill-annealed type 316 LN SS material was used for the fatigue tests. The 25 mm thick plate from which the specimens were machined met the American Society for Testing and Materials (ASTM) specification A240-88C, and the composition and relevant physical and mechanical properties of this low-carbon, nitrogen-containing material are shown in Table 1. Additional information regarding the general microstructure, tensile properties, and strain-controlled low-cycle fatigue properties is recorded elsewhere [3].

Fatigue test specimens with a uniform-gage test section were machined parallel to the primary rolling direction of the 25 mm plate. The test specimen used for low-frequency tests ($\leqslant 10$ Hz) is shown in Fig. 1. To hold mercury around the test section of the specimen, a vial machined from commercially pure nickel was press-fit to the lower end of the specimen. The other end of the specimen was fitted with a bushing having the same outside diameter as the vial to facilitate gripping in the Instron 8500 fatigue machine. The assembly (see Fig. 2) was then ultrasonically cleaned then filled with mercury prior to being loaded in the hydraulic-actuated collet type grips of the fatigue machine.

A state-of-the-art fatigue testing system, manufactured by MTS Systems Corporation, was used to conduct high-frequency tests at 700 Hz at the University of Tennessee. To meet machine specifications, a second specimen design was required with a 5.0 mm diameter × 19 mm long uniform-gage test section and an overall length of 118.8 mm. And, consequently, a mercury containment vial was specifically designed to fit this specimen.

Table 1
Vendor ladle analysis for Jessop Steel Company heat 18474 type 316 LN stainless steel

Element	Wt%
C	0.009
Mn	1.75
P	0.029
S	0.002
Si	0.39
Ni	10.2
Cr	16.31
Mo	2.07
Co	0.16
Cu	0.23
N	0.11
Fe	Balance
Room temperature properties (test at strain rate) 8×10^{-5}/s	
0.2% offset yield strength	259.1 MPa
Ultimate tensile strength	587.5 MPa
Elongation	86.2%
Reduction in area	88.1%
Grain size	ASTM 3.7

Selected physical and mechanical properties for the mill-annealed material also included.

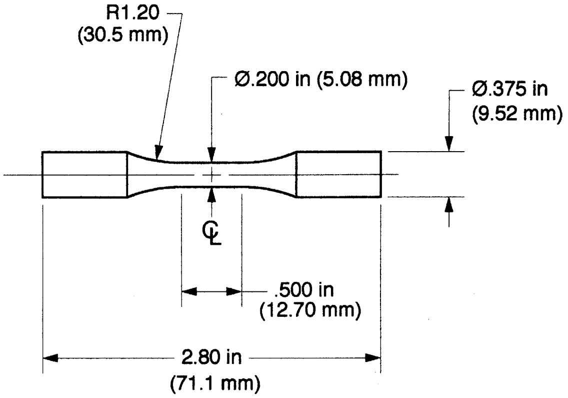

Fig. 1. Uniform-gage fatigue specimen.

2.2. Testing procedures

Constant amplitude, uniaxial load controlled fatigue tests were conducted under several loading conditions defined by R ratio where $R = S_{\min}/S_{\max}$, the ratio of the applied minimum and maximum stresses. Fully reversed tension–compression ($R = -1$) and tensile mean stress

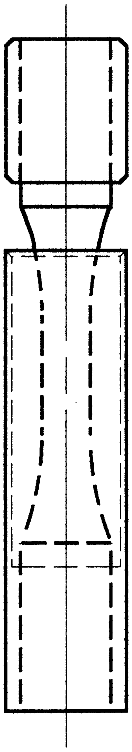

Fig. 2. Fatigue specimen with bushing and vial for containment of mercury.

($R = 0.1$) tests were conducted at room temperature, in air and mercury environments, generally employing a sinusoidal waveform over the frequency range 0.1–700 Hz. The test results were plotted as alternating stress, S_a ($S_a = (S_{max} - S_{min})/2$, one-half of the stress range) versus the number of cycles, N, to failure, generally known as a *S–N* curve.

In order to gain some insight as to the possible relationship between environment and waveshape, a limited number of low frequencies were performed at a high-stress level using two waveshapes in addition to the generally employed sine waveform. These were: (1) a general triangular waveform with equal loading and unloading ramp rates, and (2) a 'positive sawtooth' waveshape with a slow loading ramp rate for 90% of the total cycle period, followed by a rapid linear unloading segment to complete the waveform having an overall cyclic frequency of 0.1 Hz.

Initial experience in high-frequency testing of the 316 LN stainless steel in air showed that the specimen temperature would quickly rise to a steady-state temperature of 270°C during a 700 Hz fatigue initially started at room temperature with $R = 0.1$ and $S_{max} = 439$ MPa. Further, the steady-state temperature tended to decrease with decreasing σ_{max}. The observed self-heating of the specimen was likely caused by a number of inelastic effects [4] due to plastic deformation resulting from the generation and movement of dislocations, internal friction, work hardening, and the eventual formation of cracks which reach a balance with a net steady-state release of heat to the environment.

On the other hand, in mercury under the same loading condition the rise in temperature was much lower, 78°C, since the mercury actually acts as a body of cooling liquid. A slight increase in specimen temperature in mercury was not expected to show an obvious effect on fatigue life.

Generally, significant increases in temperature are known to decrease fatigue life. Consequently, in order to examine the effect of frequency on fatigue life in air and mercury at commensurate temperatures it was necessary to provide supplemental cooling for specimens tested in air. A liquid-nitrogen gas generator was employed for this purpose and thermocouples were used to monitor specimen temperature.

3. Data interpretation and evaluation

Load-cycling results provide a definition of fatigue life in terms of the applied stresses, which are described by three parameters. The range of stress, S_r, is the algebraic difference between the maximum and minimum stresses in one cycle, $S_r = S_{max} - S_{min}$. The alternating stress amplitude, S_a, is one-half of the stress range, $S_a = S_r/2 = (S_{max} - S_{min})/2$. And the mean stress, S_m, is the algebraic average of the maximum and minimum stress in one cycle, $S_m = (S_{max} + S_{min})/2$. In a completely reversed cycle test, mean stress is zero, and the ratio of the minimum stress to the maximum stress $R = S_{min}/S_{max}$ is -1. If the stress is cycled between a maximum tensile stress and no load, the stress ratio R becomes zero. And, if the stress is cycled between two tensile stresses, the R ratio is a positive number <1.

Generally, test results are plotted in terms of alternating stress amplitude versus cycles to failure (N_f), the familiar *S–N* curve. The number of cycles of stress that a metal can endure before failure occurs increases with decreasing stress. For some engineering materials, such as low-strength steel and titanium, the *S–N* curve becomes horizontal. Below this limiting stress, known as the fatigue limit or endurance limit, the material should not fail.

Other materials, including 316 stainless steel, do not exhibit a single-valued fatigue limit. Rather, the *S–N* curve continues to drop asymptotically. For such behavior, a fatigue strength for a specific number of cycles is reported, i.e., the fatigue endurance limit depends upon the required cycles.

For design purposes it is useful to know how mean stress affects the allowable alternating stress amplitude for a given life. At zero mean stress, the allowable stress

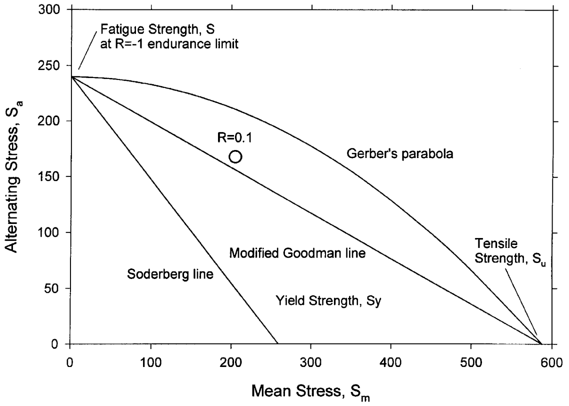

Fig. 3. Empirical diagrams showing effect of mean stress on fatigue endurance limit.

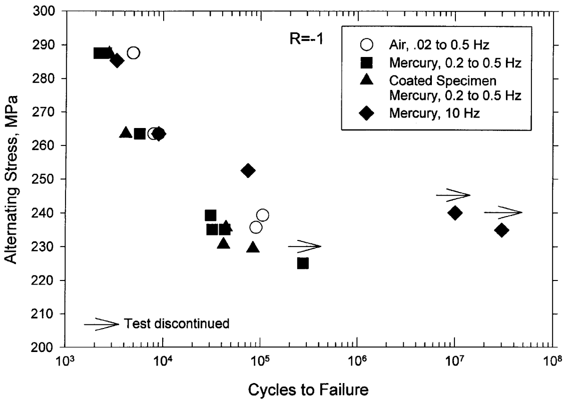

Fig. 4. Fatigue data for 316 LN stainless steel in air and mercury for $R = -1$.

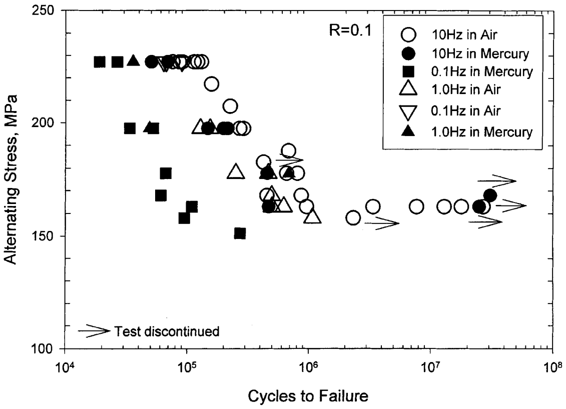

Fig. 5. Fatigue data for 316 LN stainless steel in air and mercury for $R = 0.1$.

amplitude is the effective fatigue strength for a specified fatigue life. As the mean stress increases, the permissible amplitude decreases. And, at a mean stress equal to the ultimate tensile strength of the material, the allowable cyclic amplitude is zero.

The three most widely used empirical relationships for describing the effect of mean stress on fatigue strength are shown in Fig. 3. The straight line joining the alternating fatigue strength, S, for a given life to the tensile strength, S_u of the material is the Goodman law which is, as a rule-of-thumb, applicable to brittle materials but conservative for ductile materials. Gerber's law provides a parabolic relationship between S and S_u which is generally applicable to ductile materials. And Soderberg's law described by a straight line from fatigue strength, S, to the yield strength of the material, S_y, is very conservative in most cases and is intended to be applicable to design conditions where neither fatigue failure nor yielding should occur.

In general, fatigue strength is dependent on a number of parameters including stress-cycle frequency, stress-waveshape, temperature, etc. If environment is deleterious to fatigue behavior, there can be a synergistic relationship between the environment and these parameters as well as stress ratio.

4. Results and discussion

Plots of stress amplitude versus fatigue life (*S–N* plots), Figs. 4 and 5, show results over a range of frequencies (0.1–10 Hz) in both air and mercury for R ratios of −1 and 0.1, respectively. A sharp knee in the curves is observed at approximately 1 million cycles.

Based on the available data on 30 million cycles, the *S–N* curves for $R = -1$ level off indicating a fatigue endurance limit in either air or mercury around 240 MPa, the alternating stress amplitude below which fatigue failure will not occur. Generally mercury reduced fatigue life, particularly at high-stress amplitudes (Figs. 4 and 5). However, fatigue life near the endurance limit appears to be converging for different frequencies. Comparison of data for a R ratio of −1 (fully reversed, zero mean stress load cycling) and $R = 0.1$ (tensile mean stress) shows that mean stress lowers the allowable stress amplitude for a given fatigue life, and results in lowering the endurance limit. At $R = -1$ an endurance limit of 230–240 MPa is exhibited, but the endurance limit drops to 160–170 MPa for a tensile mean stress with $R = 0.1$. This experimental observation lies between the modified Goodman and Gerber predictions shown in Fig. 3. High-cycle fatigue testing at high frequencies is continuing in order to define the fatigue limit out to 10° cycles.

At relatively low frequency, both the loading frequency and the environment (mercury) had some impact on fatigue life of type 316 LN stainless steel at high-stress levels (i.e., stresses considerable above the apparent fatigue limit). For $R = 0.1$, those data in air and mercury are plotted separately in Figs. 6 and 7, respectively, to better differentiate the effect of frequency. In air, data at 1.0 Hz showed a 30–50% reduction in fatigue lifetimes compared with results at 10 Hz. Limited testing at 0.1 Hz suggest that no further reduction in fatigue life in air would be expected at frequencies less than 1.0 Hz. At 10 Hz, fatigue lifetimes in mercury were less than those measured in air by approximately a factor of 2 at stress levels higher than the apparent fatigue limit. Furthermore, when the test frequency was lowered to 0.1 Hz, the resultant reduction in fatigue life in mercury was significantly greater than was observed in air. Fatigue lifetimes in mercury at 0.1 Hz were found to be approximately 70% lower than those at 10 Hz in mercury and nearly an order of magnitude lower than the results in air at 10 Hz.

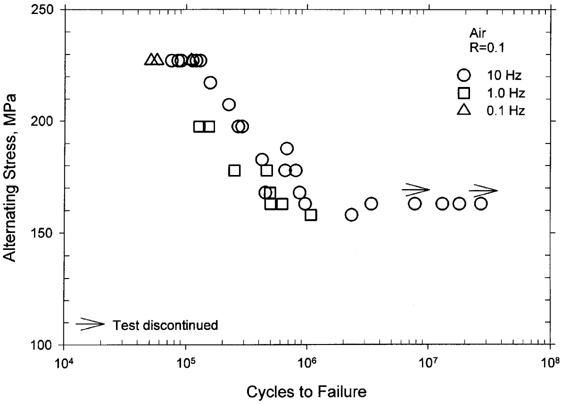

Fig. 6. Effect of frequency on fatigue life of 316 LN stainless steel in air for $R = 0.1$.

These data also suggest some effect of frequency on endurance limit. Further tests at low-stress amplitudes at such low frequencies would be very time consuming and, therefore, are not currently planned.

The process of fatigue consists of crack initiation at the surface of the test specimen, followed by progressive cyclic growth of a crack until the remaining uncracked cross-section becomes too weak to sustain the loads imposed and abrupt fracture occurs. Fracture surfaces of specimens exposed to mercury [2] were completely wetted by the mercury while the remaining surface along the specimen gage area was not wetted. Since wetting is essential to LME, this suggests that there is likely no effect of mercury on crack initiation, but mercury apparently enhances crack propagation rates, and consequently lowers fatigue life.

A few fracture surfaces were examined earlier [2] in a scanning electron microscope (SEM). For a high-stress amplitude, the specimen tested in mercury appeared to be much more brittle with widespread intergranular cracking, suggesting that liquid metal embrittlement is occurring in mercury. At a low-stress amplitude near the fatigue endurance limit, fracture surfaces in mercury and air became more similar.

In fatigue crack growth tests on 316 stainless steel in air at 538°C [5], it has been observed that at frequencies in the range 0.0067–6.67 Hz, crack growth rate increased as the frequency decreased. Since wetting by mercury is likely to be enhanced in a freshly formed crack, it might be expected that liquid metal embrittlement effects would result in a more significant (synergistic) frequency effect in mercury compared with air. The frequency effect on fatigue life over the range 0.1–10 Hz is more promounced in mercury (Fig. 7) than in air (Fig. 6). However, between 10 and 700 Hz the effect nearly disappears as shown in Fig. 8 when test temperature (self-heating) at high frequency was controlled. However, future crack

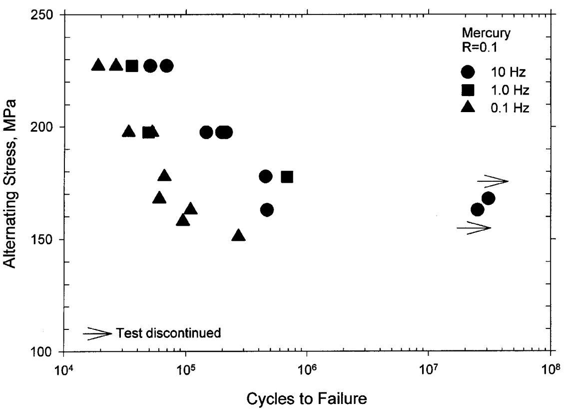

Fig. 7. Effect of frequency on fatigue life of 316 LN stainless steel in mercury for $R = 0.1$.

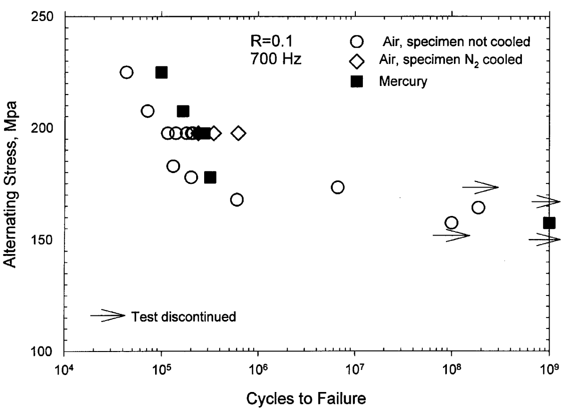

Fig. 8. Effect of mercury environment on the fatigue life of 316 LN SS at 700 Hz.

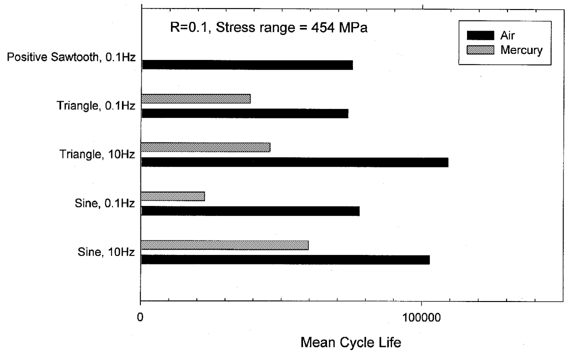

Fig. 9. Comparisons of fatigue life of 316 LN stainless steel in air and mercury employing various waveforms.

growth rate testing of 316 LN stainless steel (the SNS target vessel material) in mercury is being considered.

When environment has an impact on fatigue crack growth propagation, waveform (i.e., the shape of the load cycle) may be another important loading variable (in addition to frequency) since it can have the effect of extending the time the crack is open during the crack extension (loading) portion of waveform as well as increasing the time the crack tip stress intensity factor exceeds a threshold value, K_{th}, required for the crack to propagate. For example if K_{th} occurred when the load reached 0.75 S_{max}, then sinusoidal and positive sawtooth waveforms with the same frequency would result in twice as much time for environmental interaction compared with a triangular waveform. At lower threshold values, the reaction time offered by positive sawtooth waveform would be significantly higher than either the sinusoidal or triangular waveshape. In Fig. 9 fatigue lives at 10 Hz in air for replicate tests at a high-stress range with $R = 0.1$ were comparable for sine and triangular waveforms. Similarly, fatigue lives at 0.1 Hz in air were comparable for all three waveforms, though a frequency effect compared with 10 Hz is evident. In mercury the reduction in fatigue life at 10 Hz was comparable for sine and triangular waveforms. However, the sinusoidal waveshape resulted in a significantly lower fatigue life compared with the triangular waveform. Tests in mercury employing a positive sawtooth waveform are currently in progress.

5. Summary

S–N fatigue data have been obtained for 316 LN stainless steel at room temperature in air and mercury for two loading conditions, including fully reversed, zero mean stress $R = -1$ and tensile mean stress with a minimum/maximum load ratio (R) of 0.1. Post-test metallographic examination of specimens tested at high stresses shows evidence of liquid metal embrittlement. More intergranular cracking was observed in mercury than in air. Fatigue crack initiation was similar in each environment, but there was less resistance to crack propagation in mercury. For load-cycle frequencies over the range 0.1–10 Hz, mercury was deleterious to fatigue life. Generally, as frequency decreased, fatigue life decreased in the low-cycle region. However, fatigue life near the endurance limit in the high-cycle region appears to converge for the different frequencies. As expected, mean stress reduces the endurance limit but the effect may be accentuated in mercury. Limited testing at 700 Hz shows similar fatigue lives in air and mercury suggesting that there is a critical range of frequencies in which LME in mercury is significant. Future investigation of crack growth propagation rates in 316 LN stainless steel may reveal synergistic relationships of the mercury environment with load ratio, crack-tip stress intensity factor range, frequency, waveform, and temperature.

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