Environmental Degradation of 316 Stainless Steel in High Temperature Low Cycle Fatigue

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SUMMARY

Procedures based on modification of the Conventional Strainrange Partitioning method are proposed to characterize the time-dependent degradation of engineering alloys in high-temperature, low-cycle fatigue. Creep-fatigue experiments were conducted in air using different waveforms of loading on 316 stainless steel at 816 °C (1500 °F) to determine the effect of exposure time on cyclic life. Reductions in the partitioned cyclic lives were observed with an increase in the time of exposure (or with the corresponding decrease in the steady-state creep rate) for all the waveforms involving creep strain. Excellent correlations of the experimental data were obtained by modifying the Conventional Strainrange Partitioning life relationships involving creep strain using a power-law term of either (1) time of exposure or (2) steady-state creep rate of the creep-fatigue test. Environmental degradation due to oxidation, material degradation due to the precipitation of carbides along the grain boundaries and detrimental deformation modes associated with the prolonged periods of creep were observed to be the main mechanisms responsible for life reductions at long exposure times.

INTRODUCTION

It is now widely accepted that the creep-fatigue life of materials at elevated temperatures is influenced by the cyclic deformation modes as well as the time of exposure to the environment. The waveform of loading dictates the cyclic deformation processes in a material, and hence, affects the creep-fatigue life. The exposure, with time, of a material to interactive environments at elevated temperature can degrade its cyclic load carrying or strain absorbing capabilities due to two major reasons: (1) surface-related chemical interactions such as corrosion, oxidation, etc.; (2) bulk oxidation, and metallurgical phase instabilities. An accurate creep-fatigue life prediction procedure must incorporate, therefore, the influence of both waveform of loading and time of exposure at elevated temperature. The goal of our program was to
establish an approach for characterizing exposure time effects on creep-fatigue life at elevated temperatures. This was pursued by performing creep-fatigue experiments on 316 stainless steel at 816 °C (1500 °F) using four different types of waveforms of loading. In the following section, a background for our approach of characterizing time-dependent effects in creep-fatigue life prediction is presented. The contribution of other investigators in this area is reserved for the discussion section.

BACKGROUND

Conventional Strainrange Partitioning (CSRP) is a method developed by Manson, Halford, and Hirschberg (ref. 1) that considers two distinct deformation processes, viz., time-dependent creep and time-independent plasticity, and two directions of loading, tension and compression. CSRP predicts the creep-fatigue life at elevated temperatures by distinguishing among four different types of strainranges, $\Delta e_{pp}$, $\Delta e_{cp}$, $\Delta e_{pc}$, $\Delta e_{cc}$; each with its unique life relationship. The subscripts refer to (1) tensile plasticity reversed by compressive plasticity (pp), (2) tensile creep reversed by compressive plasticity (cp), (3) tensile plasticity reversed by compressive creep (pc), (4) tensile creep reversed by compressive creep (cc), respectively. When more than one type of strainrange is present in a hysteresis loop, life is evaluated using separate life relationships and the Interaction Damage Rule (ref. 2).

Time Effects in High-Temperature Fatigue

Conventional Strainrange Partitioning was proposed as a simple means to account for the influence of waveform of loading on creep-fatigue life. Originally, waveform effects on life were accounted for by considering the amount of creep strain produced per cycle, whether the creep occurred in tension or compression, and the manner in which creep is reversed by either creep or plasticity. However, no direct influence was attributed to exposure time other than its effect on inducing creep strain. A given creep strain achieved in a short time was viewed as being equally damaging as the same amount of creep strain achieved in a longer time. Since it was recognized, however, that exposure time influenced fatigue life for more reasons than governing the type of strain generated, Manson (ref. 2), Halford, Saltsman, and Hirschberg (ref. 3) later attempted to incorporate indirectly the additional time effects by introducing time-dependent creep ductility, $D_c$, into the coefficients of the Strainrange Partitioning life relationships. While this procedure yielded appropriate corrections for exposure time, it was based on a monotonic property not involving cyclic deformation. It was, therefore, considered appropriate to investigate a form of correction based on cyclic properties and which directly involved exposure time or creep rate as the variables. This report describes the results of our program.

Steady-State Creep Rate

Priest and Ellison (ref. 4) working with 1 CrMoV steel, utilized creep strain rate to modify their CP and PC life relationships. Their rationale was that the nature of microscopic damage introduced would be dependent upon the rate at which creep is introduced. (Alternatively, they could have used exposure time since it is approximately inversely related to the creep rate.)
Improved life predictions were reported by Prlest and Ellison by dividing the CP strainrange into components of CPm (matrix dominated), CPw (wedge dominated), and CPR (cavity dominated); and the PC strainrange into PCm (matrix dominated) and PCw (wedge dominated), and generating a separate life relation for each component strainrange. The time dependency of CC strainrange was not addressed by them. In practice, their subdivision of strainranges is complex. Hence, a simpler and more practical approach is desirable. We have accomplished this in the present paper by incorporating the steady-state creep rate directly into the CP, PC, and CC life relationships. The resulting life relations are termed as "Steady-State Creep Rate Modified Strainrange Partitioning Life Relationships." An alternate modification of the Strainrange Partitioning life relationships according to the total exposure time of the creep-fatigue test has also been examined, as discussed in the next section.

Exposure Time

Manson and Zab (refs. 5 and 6) proposed several forms of ductility variations with exposure time to model the environmental effect on creep-fatigue life when the life relationships are expressed in terms of ductility. They also suggested that a time-dependent term be included directly in the Strainrange Partitioning life relationships to account for the effects of environment on creep-fatigue life. However, no experiments were conducted to verify the relations. Ellison (ref. 7), working with 1CrMoV steel, reported improved CP life predictions by utilizing a modification of the CP life relationship based on the hold-time of the cycle. The exposure time effect on the PC and CC strainranges was not reported. In this paper, the exposure time in a creep-fatigue test is directly incorporated into the CP, PC, and CC life relationships. The resulting life relationships are termed as "Exposure Time Modified Strainrange Partitioning Life Relationships." The Steady-State Creep Rate- and Exposure Time-Modified Strainrange Partitioning life relationships are termed as "Generalized Strainrange Partitioning Life Relationships."

EXPERIMENTAL RESULTS

Creep-fatigue experiments were conducted in air at 816 °C (1500 °F) on solid hourglass specimens of 316 stainless steel using a servo-hydraulic fatigue testing machine. Typically 316 stainless steel is used in the nuclear electric power industry as a structural material around 538 to 704 °C (1000 to 1300 °F). However, a temperature of 816 °C (1500 °F) was chosen intentionally for this study to accelerate the material degradation under creep-fatigue conditions. Thus, significant reductions in creep-fatigue life could be observed when the exposure times were increased from a few hours to about 300 hr. The experimental procedures, equipment details, and tabular data are presented in reference 8. Representative hysteresis loops used in this study are shown schematically in figure 1. In the case of CP, PC, and CC experiments, tensile and compressive creep strains were introduced by stress-hold periods. Different exposure times were achieved by using different creep stress levels. In all the experiments involving creep strain, the ratio of total creep strain (transient + steady state) to the total inelastic strain was controlled at a nominal value of 0.6. This condition was most readily achieved by the selection of the hysteresis loops shown in figure 1. Failure of a specimen was taken to be complete separation into two pieces. The fatigue life results of
the strain-controlled PP experiments conducted with 0.2 Hz sinewave are shown in Figure 2. The life relation shown represents the least squares fit of the data points. The strainrange exponent of -1.60 is typical of austenitic stainless steels at elevated temperatures (ref. 1).

In the case of CP, PC, and CC experiments, each stabilized hysteresis loop (near the half-life) was partitioned considering only the steady-state portion of the creep strain. Transient creep strain was considered as plastic strain (refs. 8 and 9). The Interaction Damage Rule (ref. 2) was used together with the PP life relation to obtain the partitioned cyclic life for each test. The partitioned life is referred to as the "analytically observed" life (N0BS). In all the three cases, the partitioned cyclic lives decreased by a factor of about 2 as the time of exposure of the creep-fatigue test increased by a factor of 10. The exposure times of the creep-fatigue experiments ranged from a few to several hundred hours. The life relationships were established for each strainrange type utilizing all the data of that type with various exposure times.

The Conventional Strainrange Partitioning life relationships follow the Manson-Coffin law (refs. 1 and 2). To take exposure time effects into account, two separate general forms of modifications were added to the three life relationships of the strainranges involving creep strain. A power-law in either steady-state creep rate or exposure time was utilized to modify the conventional life relationships of the strainranges involving creep strain. The general form of the life relationships utilized in this study for the strainranges involving creep strain are given below:

\[ N_{ij} = A(\Delta \epsilon_{ij})^\alpha \phi^\beta \]  

where

1. \( \epsilon \) (plasticity), c (creep)

\( N_{ij} \) is the partitioned cyclic life

\( \Delta \epsilon_{ij} \) is the generic strainrange which is identically equal to the total inelastic strain range of the hysteresis loop, \( \Delta \epsilon_{in} \).

\( \phi \) is either a parameter involving steady-state creep rate, \( \epsilon_{ss} \) or exposure time, \( t_e \).

A, \( \alpha \) and \( \beta \) are constants that are evaluated individually for each life relationship using regression analysis.

The Conventional Strainrange Partitioning life relationships and the Generalized Strainrange Partitioning life relationships, generated by linear and multiple linear regression analysis, respectively, are as follows:
Conventional Strainrange Partitioning Life Relationships

\[ N_{pp} = 0.521(\Delta \varepsilon_{pp})^{-1.60} \]
\[ N_{cp} = 0.144(\Delta \varepsilon_{cp})^{-1.33} \]
\[ N_{pc} = 5.86(\Delta \varepsilon_{pc})^{-0.837} \]
\[ N_{cc} = 3.83(\Delta \varepsilon_{cc})^{-1.01} \]

where \( N_{cp}, N_{pc}, \) and \( N_{cc} \) are generic cyclic lives and \( \Delta \varepsilon_{cp}, \Delta \varepsilon_{pc}, \) and \( \Delta \varepsilon_{cc} \) are identically equal to the total inelastic strainrange \( \Delta \varepsilon_{in} \) of the hysteresis loop.

Generalized Strainrange Partitioning Life Relationships

Steady-state creep rate modified strainrange partitioning life relationships.

\[ N_{cp} = 0.646(\Delta \varepsilon_{cp})^{-1.46}(\dot{\varepsilon}_{ss,T})^{0.256} \]
\[ N_{pc} = 37.6(\Delta \varepsilon_{pc})^{-0.653} |\dot{\varepsilon}_{ss,c}|^{0.149} \]
\[ N_{cc} = 9.44(\Delta \varepsilon_{cc})^{-1.09}(\dot{\varepsilon}_{ss,ave})^{0.190} \]

where

\( \dot{\varepsilon}_{ss,T} \) is the tensile steady-state creep rate, min\(^{-1}\)
\( |\dot{\varepsilon}_{ss,c}| \) is the absolute value of the compressive steady-state creep rate, min\(^{-1}\)
\( \dot{\varepsilon}_{ss,ave} \) is \( 1/2(\dot{\varepsilon}_{ss,T} + |\dot{\varepsilon}_{ss,c}|) \)

In the CC experiments, the magnitude of the compressive creep stress was maintained at a slightly larger value than the magnitude of the tensile creep stress. This procedure was necessary to produce approximately equal magnitudes of tensile and compressive steady-state creep rates (ref. 11) and to avoid unbalanced strainranges, viz., CP and PC from being generated in a CC test. If the tensile and compressive steady-state creep rates differ significantly in a CC test, a different form of expression other than the simple average used in this study may be required.
Exposure time modified strainrange partitioning life relationships.

\[ N_{cp} = 0.113(\Delta \varepsilon_{cp})^{-1.63}(t_e)^{-0.332} \]
\[ N_{pc} = 21.8(\Delta \varepsilon_{pc})^{-0.696}(t_e)^{-0.223} \]
\[ N_{cc} = 6.72(\Delta \varepsilon_{cc})^{-1.08}(t_e)^{-0.248} \]

where \( t_e \) is the total exposure time of a creep-fatigue test in hours.

In the above life relationships the total exposure time of the creep-fatigue experiment was used. This enables a designer to determine the cyclic life of a component at any strainrange and time of exposure. It should be noted that average time per cycle could also have been used instead of total exposure time to characterize the time effects. The resulting life relationships would be very similar to those obtained using total exposure time.

The results of the CP tests are presented in reference 10, and are repeated here for completeness and comparison with the results of PC and CC strainranges. Comparison of the "analytically observed" cyclic lives \( N_{obs} \) and "correlated" \( N_{corr} \) cyclic lives is presented in figure 3 for the Conventional, Steady-State Creep Rate (SSCR) Modified and Exposure Time (ET) Modified Strainrange Partitioning (SRP) life relationships, respectively. For all three strainrange types, Conventional SRP is slightly unconservative at the longest exposure times (and over conservative at the short exposure times). The SSCR- and ET-Modified SRP life relationships correlate the data better than Conventional SRP, being within a factor of 1.5 for each generic strainrange. This clearly shows that some of the "scatter" in the Conventional Strainrange Partitioning results of figure 3(a) is due to the effect of exposure time. When this effect is taken into consideration either by including steady-state creep rate (fig. 3(b)) or exposure time (fig. 3(c)), the correlation improved significantly.

RESULTS OF METALLURGICAL ANALYSIS

To understand the underlying mechanisms of cyclic life degradation for 316 stainless steel at 816 °C (1500 °F), detailed fractographic and metallographic studies were conducted on fractured specimens of creep-fatigue experiments. Some results for CP experiments are presented in reference 10. Only major characteristic features of the PC and CC experiments are presented in this report. More complete results of the metallurgical studies are given in reference 8. In general, the fracture surfaces of the long exposure time creep-fatigue experiments are heavily oxidized, thus masking details of the fracture surface topography. The "striation-like" features observed in a short exposure time PC test and "intergranular cracking" and "attrition marks" observed in a long exposure time PC test are shown in figures 4(a) and (b), respectively. Typical oxide layer photographs of short and long exposure time CC tests taken prior to etching are presented in figures 5(a) and (b), respectively. Oxide formation is particularly severe in a long exposure time CC experiment.
AISI 316 is an austenitic stainless steel. Metallurgical instabilities exhibited by these steels are well documented in the literature (refs. 12 to 16). At elevated temperatures, when the exposure times are limited to a few hundred hours, carbide formation (M23C6) is the predominant instability. The photomicrographs in figures 6(a) and (b) illustrate carbide precipitation in short and long exposure time CC experiments. How these carbides interact with creep-fatigue loading is discussed in the next section.

DISCUSSION

Prior Methods

It is necessary to consider both the effects of frequency (or some other measure of time of exposure) and waveform of loading to accurately predict the creep-fatigue cyclic life at elevated temperatures. Creep-fatigue life prediction models that ignore waveshape effects and are based solely on frequency effects, for example, the Frequency Modified Fatigue model, reference 17, will not accurately predict cyclic lives for waveshapes not used in the evaluation of the model constants. Calculations to demonstrate this point can be found in reference 8. Attempts have been made to improve the accuracy of the Frequency Modified fatigue model by considering waveshape effects. The resultant modification is called Coffin's Frequency Separation Method (ref. 18). The waveshape effect was attributed to mean stress rather than creep-fatigue effects.

Comparison of Conventional and Generalized Strainrange Partitioning Methods

The originally proposed Conventional Strainrange Partitioning (CSRP) method models primarily the waveform effects on creep-fatigue life. Since frequency effects are not considered by CSRP, it averages the effects of exposure time over a range of creep-fatigue data. The Generalized Strainrange Partitioning (GSRP) method proposed herein models both the waveform and exposure time effects in predicting creep-fatigue life. As a result, correlations by the GSRP method are superior to those by the CSRP method. This is particularly significant for predicting creep-fatigue life at long times using short-time, creep-fatigue data. Life predictions, if based on CSRP, would be overly conservative, whereas the GSRP relations would be expected to produce more reliable predictions.

Additional Factors

In this study, cyclic life reductions of over a factor of 2 for the CP, PC, and CC life relations were observed for exposure times ranging from a few to 300 hr. Modifications of the Conventional Strainrange Partitioning life relations using a power-law of exposure time (or steady-state creep rate) has resulted in good creep-fatigue life correlations for 316 stainless steel at 816 °C (1500 °F) within the timeframe of the present experiments. However, it should be noted that different factors of life reduction might occur for time periods greater than the current range of data. Furthermore, for other materials and temperatures, a different type of modification using exposure time may be necessary to accurately correlate and predict creep-fatigue lives. The
actual micromechanisms of damage would dictate the form of modification. This report provides a specific framework for relatively short-time creep-fatigue data to be extended to longer times. Provided the damaging mechanisms do not change, the extrapolations should remain valid.

In extrapolating the time-dependencies of short-time, creep-fatigue data to the predictions of much longer times, i.e., well beyond the range of cyclic data, caution should be exercised that there are no significant changes in damage mechanism. One easy technique to follow is to examine the variation in creep-rupture ductility with rupture time in the time range of interest. Any significant changes in the slope of the ductility-time curve is a warning of a mechanism change. Techniques for how to proceed under these circumstances have not been worked out, as yet, for the present Generalized Strainrange Partitioning life relationships. However, it is suggested that many of the same techniques employed by the Ductility Normalized Strainrange Partitioning approach (ref. 3) could be applied successfully.

An issue not considered in this study is the time-dependency within the PP strainrange. The PP life relationship has not been modified in the Generalized Strainrange Partitioning life relationships. This life relationship is established by relatively high frequency strain-controlled experiments. As a result, there is very little time for either oxidation or metallurgical instabilities to degrade cyclic life in the experiments used to ascertain the PP life relation. However, when PP strainrange occurs together with CP, PC, or CC strainranges, the exposure time effect is reflected in the partitioned $N_{CP}$, $N_{PC}$, and $N_{CC}$ cyclic lives. Further research will be required to isolate any time-dependent effects on the PP life relationship from those effects on the CP, PC, and CC life relationships.

Damage Mechanisms

Oxidation. - The reductions in cyclic life at long exposure times in creep-fatigue experiments are due to creep effects, environmental interactions such as oxidation, and to metallurgical instabilities of the material. Oxidation can reduce both crack initiation and propagation lives depending upon the waveform of loading. For example, in the PC type of loading, an oxide forms almost exclusively during the compressive creep portion of the cycle. This layer grows in equilibrium with the compressive stresses present in the substrate, and, if brittle enough, can subsequently crack during the rapid excursion to the peak tensile portion of the cycle. Oxide initiated cracks can then propagate into the substrate (ref. 19). Thus, premature oxide layer cracks can reduce the crack initiation life of the substrate material. This phenomenon is more likely to occur in a long exposure time test, since sufficient amount of time is available in such a test for the oxide layer to grow. In the case of CP type loading, crack initiation and propagation lives are also likely to be reduced at long exposure times, but by different mechanisms than those in PC loading. Crack initiation may be hastened at surface connected grain boundaries where the propensity for oxidation is greatest and where larger localized creep deformation can concentrate. Thus, large localized deformation imposed on the less ductile oxidized material results in earlier localized fracturing. Liu and Oshida (ref. 20) cited the results of several investigators to illustrate that at elevated temperatures below a certain frequency, the crack propagation rate is inversely proportional to the frequency. These investigators proposed a fatigue crack growth model based on intermittent microruptures of
grain boundary oxide to explain this frequency dependency. Since exposure time is inversely proportional to the frequency, substantial fatigue crack propagation life reduction can occur in long-time creep-fatigue experiments. In the case of CC type loading, crack initiation and propagation lives also can be reduced at long exposure times. The mechanisms of interaction between the cyclic deformation and oxidation are combinations of those experienced in CP and PC loadings.

**Metallurgical instabilities.** - Metallurgical instabilities, such as carbide precipitation in austenitic steels, can alter deformation behavior, and hence, can influence creep-fatigue response. In 316 stainless steel at 816 °C (1500 °F), under zero mechanical load, the carbide formation occurs predominantly along the grain boundaries. As the exposure time is increased, grain boundary "coarsening" occurs (ref. 8). When mechanical load in the form of creep is applied, these grain boundary carbides resist the movement of grain boundaries thereby increasing the local stresses. Creep deformation is achieved by fracturing the grain boundary carbides, thus creating intergranular cracks, triple point wedge cracks, etc. These cracks occur not only at the surface, but also within the bulk of a material, thus reducing its cyclic crack initiation and propagation lives. Since carbide precipitation increases with exposure time, significant life reduction can occur at longer exposure times in creep-fatigue experiments.

Creep-fatigue life can also be reduced due to interaction between oxidation and metallurgical instabilities. It should be realized, however, that the exact mechanisms depend upon the metallurgical state of a material as well as the type of the environment. In order to predict the creep-fatigue life in a reliable manner, it is essential to consider material degradation mechanisms appropriate to the material/environment system in which the actual component is to operate.

**CONCLUSION**

The Conventional Strainrange Partitioning life relationships which model the effect of waveform of loading on creep-fatigue life are generalized to include the effect of exposure time. This is accomplished by modifying the Conventional Strainrange Partitioning life relationships using either Steady-State Creep Rate (SSCR) or Exposure Time (ET). The resulting SSCR- and ET-Modified Strainrange Partitioning life relationships correlate the creep-fatigue experimental data of 316 stainless steel at 816 °C (1500 °F), significantly better than the Conventional Strainrange Partitioning life relationships. The reductions in the cyclic life at long exposure times are likely due to oxidation and precipitation of carbides.

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REFERENCES


FIGURE 1. - HYSTERESIS LOOPS UTILIZED IN DEVELOPING THE GENERALIZED STRAINRANGE PARTITIONING LIFE RELATIONSHIPS.

FIGURE 2. - PP LIFE RELATIONSHIP, 316 SS AT 816 °C (1500 °F).
Figure 3. Comparison of observed and correlated cyclic lives.

(A) Life correlations by conventional strainrange partitioning method.

(B) Steady state creep rate modified strainrange partitioning method.

(C) Exposure time modified strainrange partitioning method.

Figure 3. Concluded.
FIGURE 4. - FRACTOGRAPH OF 316 STAINLESS STEEL FAILED AT 816 °C (1500 °F) UNDER PC LOADING.

(A) "STRIATION-LIKE" FEATURES, \( t_e = 3.91 \) HOURS.

(B) INTERGRANULAR CRACKING AND "ATTRITION MARKS", \( t_e = 87.72 \) HOURS.

FIGURE 5.- OXIDE LAYER FORMATIONS ON SPECIMENS OF 316 STAINLESS STEEL FAILED AT 816 °C (1500 °F) UNDER CC LOADING.

(A) EXPOSURE TIME, \( t_e = 4.51 \) HOURS.

(B) EXPOSURE TIME. \( t_e = 224.03 \) HOURS.
FIGURE 6. - PRECIPITATION OF CARBIDES IN CC SPECIMENS OF 316 STAINLESS STEEL FAILED AT 816 °C (1500 °F) UNDER CC LOADING.
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Procedures based on modification of the Conventional Strainrange Partitioning method are proposed to characterize the time-dependent degradation of engineering alloys in high-temperature, low-cycle fatigue. Creep-fatigue experiments were conducted in air using different waveforms of loading on 316 stainless steel at 816 °C (1500 °F) to determine the effect of exposure time on cyclic life. Reductions in the partitioned cyclic lives were observed with an increase in the time of exposure (or with the corresponding decrease in the steady-state creep rate) for all the waveforms involving creep strain. Excellent correlations of the experimental data were obtained by modifying the Conventional Strainrange Partitioning life relationships involving creep strain using a power-law term of either (1) time of exposure or (2) steady-state creep rate of the creep-fatigue test. Environmental degradation due to oxidation, material degradation due to the precipitation of carbides along the grain boundaries and detrimental deformation modes associated with the prolonged periods of creep were observed to be the main mechanisms responsible for life reductions at long exposure times.

Life prediction; Creep-fatigue; Creep; Fatigue (metal); Oxidation; Strainrange partitioning; Stainless steel; Metallurgical instability; Environmental degradation

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