THE EFFECT OF MICROSTRUCTURE, TEMPERATURE, AND HOLD-TIME
ON LOW-CYCLE FATIGUE OF AS HIP P/M RENE' 95

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Summary

The effects of microstructure, temperature, plastic strain range, and hold time on the low-cycle fatigue (LCF) life were studied for Rene' 95, an important Ni base superalloy used in jet engine disks. It was shown that the life could be varied by approximately an order of magnitude at elevated temperatures by simple heat treatments. The life was largest for the microstructure that promoted the most homogeneous deformation mode. The results are explained using the concept of a synergistic interaction between the deformation mode and boundary oxidation.

Introduction

Further improvement in the performance of aircraft jet engines depends on the development of new materials with improved strength/weight ratios and enhanced fatigue and creep properties and oxidation resistance at high temperatures. Consequently, new alloys and processes are being developed for such applications. Powder Metallurgy (P/M) is becoming increasingly important in the manufacture of critical components, such as turbine disks, where it is essential to overcome the problems of macrosegregation; poor workability; and to conserve materials that are in short supply. Rene' 95 is a high strength P/M superalloy developed for use in compressor and turbine disks operating at temperatures up to 649 C (1200 F) (1).

In the push for improved performance, components are being subjected to increasingly severe thermal, chemical, and mechanical environments. Given this situation, it is desirable to identify low-cycle fatigue (LCF) damage processes as a function of microstructure, temperature, strain range, and environment. Such information is indispensable in developing new materials and in more appropriately applying existing ones. With such goals in mind, a study was undertaken to examine the effects of these important variables on the LCF behavior of As-HIP P/M Rene' 95.

This research was conducted to evaluate and understand the LCF behavior of As-HIP Rene' 95 in three different microstructural conditions in the temperature range of 25 to 871 C (70 to 1600 F). The effect of hold time at 649 C (1200 F), the maximum disk operating temperature, was also studied.

Materials and Experimental Procedure

As-HIP Rene' 95 in the form of two 2.54-cm thick and 15.24-cm diameter disks was the starting material. These disks were cut from a cylindrical compact 15.24 cm in diameter and 45.7 cm long. The pre-alloyed powder used was -60 mesh and was produced by an argon atomization process. The HIP parameters for the compacts were 1120 C (2048 F) for 3 hours at a pressure of 103 MPa. The chemical composition of the alloy is given in Table I. The heat treatment was varied to obtain different γ' sizes and grain sizes designated heat treat

Table I. Chemical Composition (Wt%) of Rene' 95 Used in This Study

C	Mn	Si	S	Cr	Co	Mo	Fe
0.05	<0.01	0.08	0.005	12.86	8.28	3.53	0.05

Ta	Cb	Zr	Ti	Al	B	W	Ni
<0.01	3.50	0.04	2.49	3.61	0.0009	3.42	Bal

A, B, and C. The goal of the different heat treatments was to obtain microstructures having different slip behavior. Heat treatment A is a commercial process while heat treatment B results in a structure similar to that used in another study (2) in which superior crack propagation behavior was observed. Heat treat C was used to obtain the finest grain size and largest γ' size. Details of the heat treatments and the resultant microstructures are given in Table II. Longitudinal strain controlled LCF tests were conducted using a closed-loop servohydraulic test system. All continuous cycling tests were performed in a fully reversed mode ($R_{\epsilon} = -1$, $A_{\epsilon} = \infty$, $\nu = 0.33$ Hz) using a triangular waveform. Hold time tests were conducted using a 10-900-10 second sequence with the hold at maximum strain. Induction heating was utilized for all high temperature tests. Test results are plotted in Fig. 1 and 2. Detailed optical, scanning electron microscopy (SEM) and transmission

Table II. Heat Treatments Used in This Study

MATERIAL	SOLUTION TREAT	PRECIPITATION AGING	RESULTANT MICROSTRUCTURE
RENE' 95 (AS-HIP)			
"A" HEAT TREAT	1149°C(2100°F)/1 HR. IN VACUUM. QUENCHED IN SALT BATH TO 538°C(1000°F). AC.	871°C(1600°F)/1 HR PLUS 649°C(1200°F)/24 HRS. AC	GRAIN SIZE = 5-50 μ γ' SIZE = 0.2 μ
"B" HEAT TREAT	1191°C(2175°F)/4 HRS. PLUS 1149°C(2100°F)/1 HR. IN VACUUM. OQ	871°C(1600°F)/1 HR. BQ.	GRAIN SIZE = 30-50 μ γ' SIZE = 0.08 μ
"C" HEAT TREAT	1149°C(2100°F)/1 HR. IN VACUUM. OQ.	982°C(1800°F)/72 HRS. BQ.	GRAIN SIZE = 5-10 μ γ' SIZE = 0.5 μ
AC = AIR COOLED, OQ = OIL QUENCHED, WQ = WATER QUENCHED, BQ = BRINE QUENCHED.

Figure 1 - Coffin-Manson representation of the fatigue behavior of As-HIP Rene' 95 in Condition A. $T = 649^\circ\text{C}$, $R_\epsilon = -1$, $\nu = 0.33\text{ Hz}$

Figure 2 - Comparison of low-cycle fatigue as a function of temperature of As-HIP Rene' 95 for conditions A, B, and C. $R_\epsilon = -1$, $\Delta\epsilon_p = 0.16\%$, $\nu = 0.33\text{ Hz}$.

electron microscopy (TEM) analyses were conducted to fully characterize initial microstructures, crack initiation, fracture mode, and deformation substructures.

Discussion of Results

Microstructures

The microstructures of all three conditions are shown in Fig. 3. For Condition "A" the grain size range was 5 to 50 μ and the aging γ' was about 0.2 μ . There were also some intermediate sized γ' ($\sim 2 \mu$). In addition to intermediate γ' , larger γ' precipitates were observed at grain boundaries. These remained unsolutioned, grew during subsequent heat treatment and had the effect of limiting the grain size. Interfacial dislocations were not observed on the fine matrix γ' , indicating complete coherency between the matrix and the precipitate for γ' sizes up to about 0.2 μ . For Condition "B" the grain size range was 30 to 50 μ with γ' sizes of about 0.08 μ . Areas close to the grain boundaries (Fig. 3B) were lean in γ' . This is the result of the formation and growth of grain boundary γ' which causes a nearby γ' depleted zone. The formation of grain boundary γ' and associated depleted zone is related to diffusion of Al, a strong γ' former, to the grain boundaries. For Condition "C", the grain size was extremely fine and averaged about 5 to 10 μ . Intermediate γ' (2 μ) and fine γ' (0.5 μ) were observed within the grains. Similar to Condition A, interfacial dislocations were not seen on the fine γ' .

Heat treatments A and B had essentially the same grain size and could be used to explore the effect of γ' size where previous studies (2) have suggested a transition in the deformation mode from looping at 0.2 μ to shearing at 0.08 μ . The effect of grain size at comparable γ' sizes could be studied by comparing treatments A and C.

These microstructures were expected to provide a fairly broad range of slip behavior with Condition B showing the least homogeneous slip (presumably beneficial for crack propagation resistance (2,3) and Condition C showing the most homogeneous slip (presumably beneficial for crack initiation resistance).

Low-Cycle Fatigue

The continuous cycling and hold time LCF behavior at 649 C (1200 F) for Condition A is shown in Fig. 1. The apparent scatter is reduced by using a bilinear fit. The implication of a two-segment curve is that different damage mechanisms are operating at low and high strains (4). For tests conducted with a 15-minute dwell at maximum strain, the fatigue life was comparable to that for continuous cycling. This behavior is discussed in a later section. Test results for Condition A conducted at constant $\Delta\epsilon_p = 0.16\%$ over the temperature range of 25 to 871 C (70 to 1600 F) are plotted^p in Fig. 2. The life is seen to decrease rapidly above about 200 C. The data for Conditions B and C is also shown in Fig. 2 and for Condition C the life also decreases rapidly above 200 C. In all three conditions, the LCF life decreased as the temperature increased, indicating that creep and/or environmental effects became increasingly important. Figure 2 clearly shows that the LCF life for Condition C is superior to the other conditions over the temperature range 25 to 649 C (70 to 1200 F). Material in Condition B exhibited the shortest life. The superior fatigue resistance of Condition C is discussed below in terms of deformation mode and environmental attack.

CONDITION A

CONDITION B

A

B

Figure 3 - Microstructures of the three heat treated conditions of As-HIP Rene[®] 95 used

SEM Analysis of Fracture Surfaces

Condition A

For tests conducted at a plastic strain range of 0.16% and at different temperatures, cracks always originated at the surface, either by a Stage I crystallographic mechanism or at a surface connected pore. A typical initiation site is shown in Fig. 4A. Extensive surface microcrack formation on slip bands is shown in Fig. 4B. Similar behavior in other superalloys has also been reported by other researchers (5). At high temperatures (649 C), cracks initiated at surface pores, propagated initially by a Stage I mode and then became intergranular as shown in Fig. 5.

SPECIMEN NO. UC-25, $\Delta\epsilon_p = 0.16\%$, $N_f = 9657$ CYCLES

Figure 4 - SEM fractograph of As-HIP Rene' 95 (condition A) tested at 25 C (77 F). (A) stereo pair showing stage I crack initiation. (B) microcracks are seen forming at slip extrusions on the specimen gage section surface.

For the continuously cycled specimens tested at 649 C (1200 F), it was found that regardless of the strain level fatigue cracks initiated from pores located within approximately one pore diameter of the surface (Fig. 6). Since the initiation sites were similar in all cases, the data shown in Fig. 1 can be analyzed without considering complicating effects due to different initiation sites. At some point crack propagation changed from transgranular to intergranular as shown in Fig. 6. The crack length at which this transition occurs is plotted in Fig. 7, which indicates that there is a critical combination of crack length and strain at which the transition occurs. Such a parameter is related to the stress and strain field ahead of the microcrack. High stresses and strains will cause a large dilation which in turn promotes excess oxygen accumulation in the

SPECIMEN No. UC-29, $\Delta\epsilon_p = 0.16\%$,
 $N_f = 891$ CYCLES

Figure 5 - SEM fractograph of As-HIP Rene' 95 (condition A) tested at 649 C (1200 F). The crack initiated at a surface pore. Initial propagation was crystallographic followed by intergranular cracking (Ref. 7).

SPECIMEN NO. 6-1, $\Delta\epsilon_p = 0.017\%$,
 $N_f = 30,277$ CYCLES

Figure 6 - Typical crack initiation site for continuously cycled As-HIP Rene' 95 (condition A) tested at 649°C (1200°F). Surface connected pores were observed in all specimens.

boundaries. The excess oxygen will embrittle the boundaries and provide a low energy crack path which in conjunction with the high crack tip stresses causes a transition in crack propagation mechanism. The transition crack size (in microns) is given by:

$$a_h = 15.3(\Delta\epsilon_p)^{-0.23}$$

Typical SEM micrographs are shown in Fig. 8 for hold time LCF. Crack initiation occurred at the specimen surface, either at a surface connected pore, or by transgranular initiation. Specimens exhibiting pore-related initiation showed a lower fatigue life compared to those exhibiting transgranular initiation. Crack propagation in hold time fatigue was intergranular in all cases.

Figure 7 - Transition crack length as a function of plastic strain.

A

SPECIMEN NO. 9-1, $\Delta\epsilon_p = 0.211\%$,
 $N_f = 615$ CYCLES

B

SPECIMEN NO. 8-2, $\Delta\epsilon_p = 0.191\%$,
 $N_f = 798$ CYCLES

Figure 8 - Typical hold time crack initiation and propagation features shown for As-HIP Rene' 95 (condition A) tested at 649 C (1200 F). In (A) initiation took place at a surface connected pore and propagation was intergranular. In (B) initiation took place by a transgranular mechanism and the life was correspondingly increased.

Condition C

As mentioned previously, this condition showed the best LCF life at a plastic strain range of 0.16% over the temperature range 25 to 649 C (70 to 1200 F). In this condition, cracks always started in surface pores, which apparently because of the processing were present in the test specimen. At low temperatures, early propagation was by Stage I. Surface microcracks were observed on specimen gage sections, and these cracks were related to shear bands (Fig. 9). At high temperatures, cracks originated at surface pores and propagated intergranularly, presumably due to environmental attack, Fig. 10 (6).

SPECIMEN NO. UC-35, $\Delta\epsilon_p = 0.16\%$, $N_f = 3170$ CYCLES

Figure 9 - SEM fractograph of As-HIP Rene 95 (condition C) tested at 427 C (800 F). (A) crack initiated at a surface pore. A clear transition from initial Stage I to Stage II propagation is seen. (B) specimen gage section surface shows microcracks forming at many slip bands.

SPECIMEN NO. UC-36, $\Delta\epsilon_p = 0.16\%$, $N_f = 1545$ CYCLES

Figure 10 - SEM fractograph of As-HIP Rene 95 (condition C) tested at 649 C (1200 F). (A) crack initiated at a surface pore and initial propagation was Stage I transgranular. In (B) an area slightly ahead of that in (A) is shown. Note intergranular crack propagation.

Condition A

At low temperatures (<426 C/800 F) and constant $\Delta\epsilon_p$ (0.16%), as well as at low strains and high temperature (649 C/1200 F), slip was always concentrated in discrete bands as shown in Fig. 11 and 12. Figures 11B and 12B show γ' precipitate shearing on [111] planes by use of a dark field image in which locally disordered (sheared) γ' exhibits an absence of diffraction contrast. Many slip bands appear to have originated at grain boundary carbides.

Figure 11 - TEM micrograph of As-HIP Rene 95 (condition A) tested at 25 C (77 F). (A) bright field image using [002] reflection showing planar slip. (B) dark field image using [001] superlattice reflection of the area in (A). Note precipitate shearing (arrow).

Figure 12 - Deformation character for As-HIP Rene 95 (condition A) tested at 649 C (1200 F). (A) bright field image using [020] reflection shows slip bands. (B) dark field image of (A) using [010] reflection. Note shearing of γ' (Ref. 7).

It was also frequently observed that the deformation bands were associated with the large or intermediate size γ' particles as seen in Fig. 12 and 13. The implication is that there were high stresses associated with the large γ' particles which were sufficient to initiate the deformation process or

SPECIMEN NO. UC-6, $\Delta\epsilon_p = 0.16\%$, $N_f = 1164$ CYCLES

Figure 13 - TEM micrograph of As-HIP Rene 95 (condition A) tested at 427 C (800 F). (A) bright field image using [200] reflection. Deformation occurred on {111} slip planes. (B) dark field image of (A) using [100] superlattice reflection, shows shearing of γ' .

that interfacial dislocations on the γ' particles acted as dislocation sources for the deformation process (7).

At high temperatures (649 C/1200 F) and high strains, deformation was much more homogeneously distributed (Fig. 14). For these conditions, it is clear that more bowing is taking place and that dislocations appear to be stored on the faces of large γ' particles. Of course, the shearing seen in Fig. 12 would also be taking place, although the discrete slip bands were obscured by the high dislocation density. Deformation appeared to involve a mixture of mechanisms at this temperature and the observations are consistent with an increasing amount of thermally activated deformation (e.g., climb, thermally activated cross slip) compared to the lower temperatures.

SPECIMEN NO. UC-29, $\Delta\epsilon = 0.16\%$,
 $N_f = 891$ CYCLES

An explanation of the bi-linear nature of the Coffin-Manson curve shown in Fig. 1 can be offered based on the TEM results. For high strains, the internal stresses are large and homogeneously distributed. Consequently, there will be high average boundary stresses which under appropriate conditions will give rise to boundary failure in either the initiation or propagation process. On the other hand, at low strains, deformation was restricted to a few slip bands and even though the stresses at the tip of the slip band are high, the probability of encountering an embrittled region was low. There was thus a tendency for slip to transfer from one grain to another without opening up a boundary crack. Furthermore, when slip is planar and contained in a small number of bands, it should be more reversible and the rate of damage accumulation correspondingly low. These factors favor transgranular cracking and an increase in

fatigue life. Thus, the low strain life is longer than that which would have been predicted by simple extrapolation of the high strain behavior.

The substructure associated with hold time is shown in Fig. 15. In this case, the dislocation density was much higher than that observed for the corresponding continuous cycling case. However, the LCF life was not significantly affected. This implies that the accumulated dislocation debris, though very high, was not sufficient in itself to initiate failure. It can be envisioned that while the deformation related damage (i.e., increased dislocation density) was building up to its limiting value, the environment caused sufficient damage to initiate failure. Of course these processes may be interactive as has been suggested elsewhere (8,9,10) and the decreased stress during a hold time cycle may more than offset an increase in the amount of environmental attack.

Condition B

Condition B could not be analyzed because the specimens went into compression upon failure.

Condition C

Deformation for condition C at low temperatures occurred along discrete slip bands as shown in Fig. 16. It was very similar to condition A with the exception that there appeared to be more evidence of bypassing. Slip bands appeared to be initiated by grain boundary carbides. The SEM analysis discussed previously had shown slip bands on planes at 45 degrees to the stress axis. Thus, the discussion given for condition A at low temperatures appears to be valid for condition C and the lives were similar. At high temperature (649 C/1200 F), the deformation mode was homogeneous and a high density of dislocations were concentrated at the γ - γ' interface as seen in Fig. 17. At this temperature, dislocation climb and cross slip mechanisms appear to be dominant.

SPECIMEN NO. 11-7, $\Delta\epsilon_p = 0.059\%$,
 $N_f = 1232$ CYCLES

Figure 15 - TEM micrograph of As-HIP Rene' 95 (condition A) tested at 649 C (1200 F). Hold time test. Deformation substructure is from an area that was somewhat atypical in that the dislocation density was very high. In many grains, there was no evidence of dislocation activity (Ref. 7).

SPECIMEN NO. UC-35, $\Delta\epsilon_p = 0.16\%$,
 $N_f = 3170$ CYCLES

Figure 16 - TEM micrograph of As-HIP Rene' 95 (condition C) tested at 427 C (800 F). Bright field image showing planar slip. Note the tendency for dislocation pairing in the area at the bottom of the figure indicative of shearing and the elongated dipoles and loops indicative of a bypass mechanism.

A tentative explanation for the differences in the observed LCF life can be offered. At low temperatures, treatment A was marginally inferior to treatment C. This is probably a reflection of the fact that due to its coarser grain size and γ' size treatment C deforms more homogeneously. Thus, crack initiation would be delayed somewhat and the life would be correspondingly increased. At higher temperatures, treatment C was clearly superior to treatments A and B. Regardless of treatment, oxygen penetration along grain boundaries would be expected to be more severe, in agreement with the increased fraction of intergranular cracking that was observed. The near-surface structure was thus comprised of a set of embrittled grain boundaries and deformation was most homogeneous for treatment C and least homogeneous for treatment B. Even for inhomogeneous deformation, the probability of a slip band impinging on an embrittled grain boundary region near the surface was very high and was further increased due to the high strain and large number of slip bands. As already pointed out, the combination of high stress at the tip of the slip band and slip bands impinging on embrittled regions promotes intergranular cracking and reduces the fatigue life. In addition, results had shown that the maximum stress in a hysteresis loop at $N_f/2$ was greatest for condition B (least homogeneous deformation) and least for condition C (most homogeneous deformation)* (details are given in Ref. 6). All other factors being equal, a lower life would be expected for the highest hysteretic stress and the results are in agreement with this idea. Clearly, the high stress and inhomogeneous deformation have a synergistic effect on reducing the life. The essential features of the proposed mechanism have been recently documented for Waspaloy tested at 800 C (1450 F) and heat treated to deform by planar glide (5).

The picture that develops is that the fatigue life is determined by a complex interaction between the deformation mode and the environment. This concept has been used previously to describe the LCF behavior of other Ni-base superalloys at high temperatures (8,9).

Conclusions

1. Significant improvements in the LCF life of Rene' 95 can be obtained by microstructural control. At 649 C (1200 F) and at a plastic strain range of 0.16%, it was possible to vary the LCF life by approximately a factor of 10 with somewhat smaller (although significant) differences at lower temperatures.

*The maximum stress in a strain controlled cycle depends on the deformation mechanism. The trend of the data for our results is consistent with a maximum amount of γ' shearing for condition B with progressively less shearing and more looping for conditions A and C.

SPECIMEN NO. UC-36, $\Delta\epsilon_p = 0.16\%$,
 $N_f = 1545$ CYCLES

Figure 17 - TEM micrograph of As-HIP Rene' 95 (condition C) tested at 649 C (1200 F). Bright field image using [200] reflection shows homogeneous deformation.

2. Over the temperature range 25 to 649 C (70 to 1200 F) and at a plastic strain range of 0.16%, the longest lives corresponded to heat treatment C, which had the most homogeneous deformation and the lowest maximum stress in a fatigue cycle. On the other hand, the shortest life corresponded to treatment B, which had the least homogeneous deformation mode and the highest hysteretic stress.
3. The LCF life, especially at high temperatures, appears to be determined by complex synergistic interactions between the environment and the deformation mode.
4. At 649 C (1200 F) material in the A condition and tested at low plastic strain ranges had a longer life than would be expected by extrapolation of the high strain data. This was attributed to the small number of slip bands, slip reversibility, and low stresses.
5. The fatigue life of material in the A condition for a 15-minute hold at maximum strain at 649 C (1200 F) was at least as long as the corresponding data for continuous cycling even though the dislocation density was higher. This result is at variance with the concept of a negative creep/fatigue interaction.

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