The Influence of Hold Times on LCF and FCG Behavior in a P/M Ni-Base Superalloy

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Summary

The relative importance of creep and environmental interactions in high temperature fatigue behavior has been investigated for as-HIP René 95. Strain-controlled low cycle fatigue and load-controlled fatigue crack growth tests were performed at elevated temperatures in argon, followed by fractographic analyses of the fracture surfaces by scanning electron microscopy. Fatigue lives were drastically reduced and crack growth rates increased one hundred fold as a result of superposition of hold times on continuous cycling. A change in fracture mode with hold time also was noted. Chromium oxide was detected on the fracture surface by Auger electron spectroscopy. The drastic changes in fatigue resistance due to hold times were attributed primarily to environmental interactions with fatigue processes.

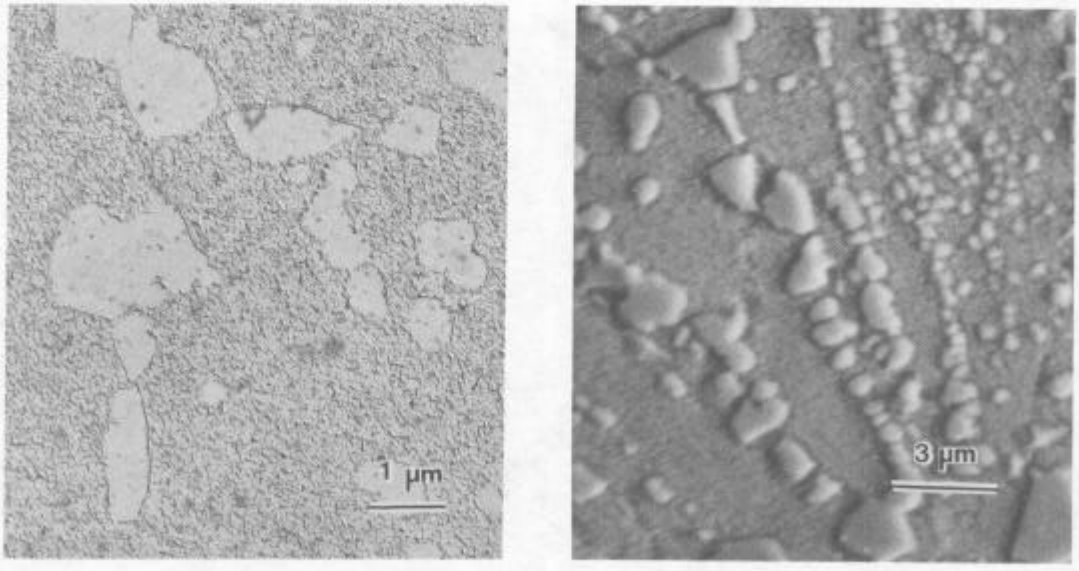
Introduction

Hot isostatically pressed (HIP) nickel-base superalloys are of considerable interest because of their near net shape processing capability and substantial cost reduction. Fatigue crack initiation in these alloys invariably occurs at some preexisting defect, usually an inclusion or pore(1,2). However, elevated temperature fatigue resistance is predominantly determined by crack growth once the crack is initiated(3). The fatigue resistance is limited due to simultaneous occurrence of time dependent processes, notably creep and oxidation(4,5). The purpose of this investigation was to examine the effects of creep-fatigue interactions on cyclic crack growth behavior of René 95 with reduced environmental interactions. Both strain-controlled low cycle fatigue (LCF) and load-controlled fatigue crack growth (FCG) experiments were carried out to characterize the fatigue behavior.

Results obtained in this study are compared to those obtained by other investigators in different microstructural conditions (e.g. HIP + forged) and other environments. The data are analyzed to isolate creep or oxidation effects in order to establish the most plausible mechanism of time-dependent damage associated with cyclic crack growth.

Material

The composition and representative mechanical properties of René 95 are listed in Table I. The alloy was produced by hot isostatically pressing the argon atomized -60 mesh powder. Prior to heat treatment 25mm thick disks were cut from as-HIP cylinders. The HIP cycle was 103.5MPa, 1121°C, 3 hrs. Heat treatment was carried out at 1150°C for 1 hr, quenched to 538°C, reannealed at 871°C for 1 hr, and 650°C for 24 hr, followed by air cooling. The microstructure contained different sizes of γ' precipitate. Irregularly shaped coarse γ' particles located along the grain boundaries were 1-5μm in diameter, see Fig. 1a). Fine γ' (approx. 0.25-1μm size) within the grain often showed a dendritic distribution, Fig. 1b). A small quantity of MC and $M_{23}C_6$ carbides also was observed. The fully dense alloy contained pores up to 40μm in diameter.



a) b)

Figure 1 - Electron micrographs showing: a) fine grains (size 10-15μm), two stage TEM replica b) dendritic distribution of fine γ' in coarse grains (size 40-50μm), SEM backscattered image.

Table I

Composition and Mechanical Properties of Rene' 95

Chemical Composition: wt%

C	Cr	Co	Mo	Cb	Zr	Ti	Al	B	W	Ni
0.050	12.9	8.3	3.5	3.5	0.04	2.5	3.6	.009	3.4	bal.

Mechanical Properties:

Temperature	$0.2\%\sigma_{y.s.}$ MPa	UTS MPa	RA %	El %
25°C	1215	1636	15	16
650°C	1120	1514	17	16

Experimental

Cylindrical LCF and compact tension FCG specimens were machined from peripheries and centers of the disks, respectively. Specimen surfaces were prepared by mechanical* polishing of the cylindrical gage section of LCF samples (gage length of 0.76cm and gage diameter of 0.33cm) and mechanical* polishing of CT samples. Tests were performed on a closed-loop servohydraulic fatigue testing system. CT specimens were precracked at room temperature at a frequency of 20Hz. Plane strain conditions were maintained in all FCG tests, as monitored by the ratio of plastic zone sizes to thickness.

Specimens were tested at 650°C in ultra-high purity argon in order to minimize the influence of oxidation effects, using sawtooth waveforms for continuous cycling. The argon was further purified by passing through an Oxisorb gel for FCG tests. Trapezoidal waveforms were used for creep-fatigue testing with loading and unloading rates equivalent to 0.33Hz for LCF and 1Hz for FCG and 2 min hold period at peak strain or load. Additional LCF tests were performed at 725°C to examine the influence of temperature on the time-dependent damage. A 6 min hold FCG test, aimed at determining the influence of increased creep-fatigue damage, was terminated after 5 days. The total strain range in the LCF tests was varied from 1.5% to 2.7%, with strain ratio $R=\varepsilon_{max}/\varepsilon_{min}=-1$, $A=\infty$. Since plastic strain range per cycle decreased continuously during the test, the plastic strain range at half life($N_f/2$) was used for plotting all LCF data. Some stress relaxation occurred in all tests upon each strain reversal; this was neglected in determining plastic strain range from the hysteresis loop.

Scanning electron microscopy (SEM) was utilized to identify fatigue origins and the morphology of crack growth. Failed specimens were also sectioned axially, mounted and polished to examine secondary cracks, creep cavities, if any, and then studied using optical microscopy, the SEM or two stage TEM replicas.

The dominant species present on the fracture surface were identified using Auger electron spectroscopy (AES) and the depth of the oxide layer present was determined by ion sputtering.

* to 0.3μm alumina

Results

A. LCF Behavior

The LCF data obtained at 650°C and 725°C are presented in Fig. 2. The 2 min hold at peak strain drastically reduced LCF lives. The increase in temperature did not affect the lives as severely as did the hold time. The Coffin-Manson exponent, β, that represents the slope of $\Delta\varepsilon_p$ vs N_f remained approximately constant at a value of 0.7 with or without hold time.

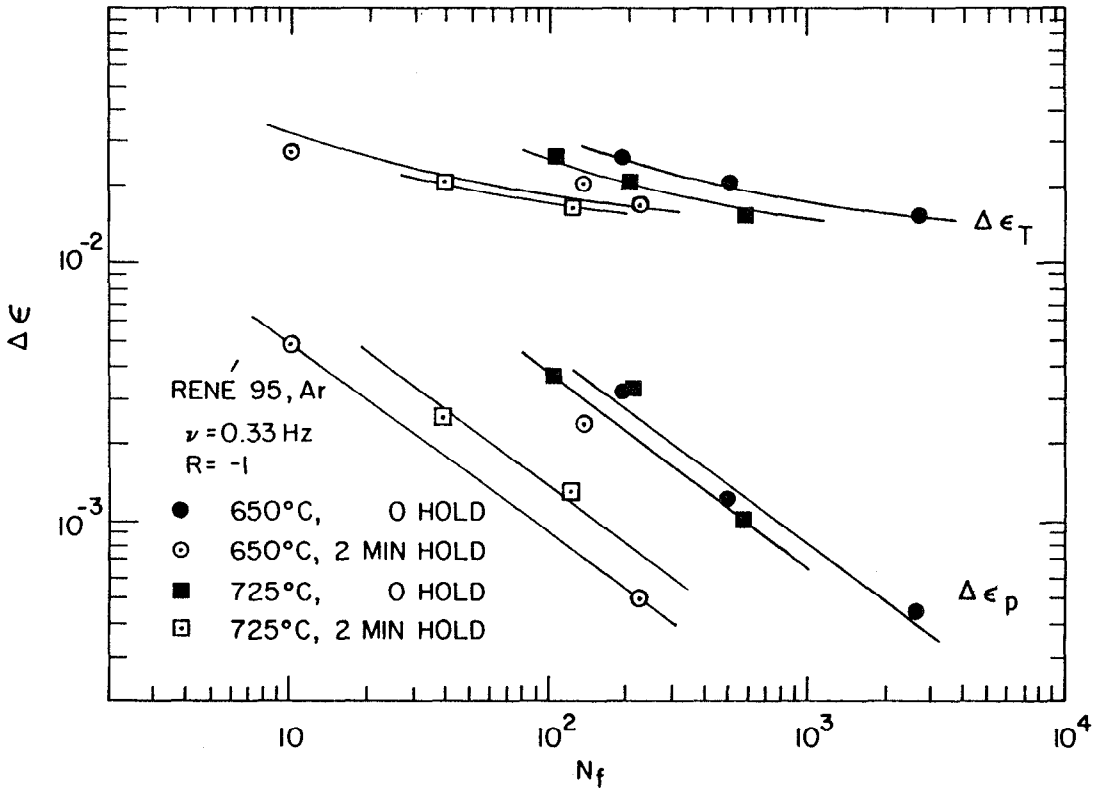


Figure 2 - LCF behavior of René 95

The LCF data of this study are compared with those of other investigators(7) in Fig. 3. The differences may be attributed to different microstructures of as-HIP and HIP + forged microstructure, different environments (air and argon) as well as differences in specimen geometries. It may be pointed out that the results of Bashir et al(6) do not show any deleterious effect on LCF life with 15 min hold in air whereas our data in argon show that even a 2 min hold has a dramatic effect in reducing life.

Crack initiation occurred near surface pores, as shown in Fig. 4a) and b). All samples showed multiple origins. The fracture mode was transgranular (TG) with continuous cycling; in the presence of hold time it changed to a mixed mode TG + intergranular (IG). Occasional fatigue striations were observed in samples with no hold times.

A comparison between fracture surfaces produced at 650°C and 725°C is shown in Fig. 5a) and b), respectively. The surface was faceted at 650°C, whereas at 725°C facets disappeared and the surface was macroscopically flat.

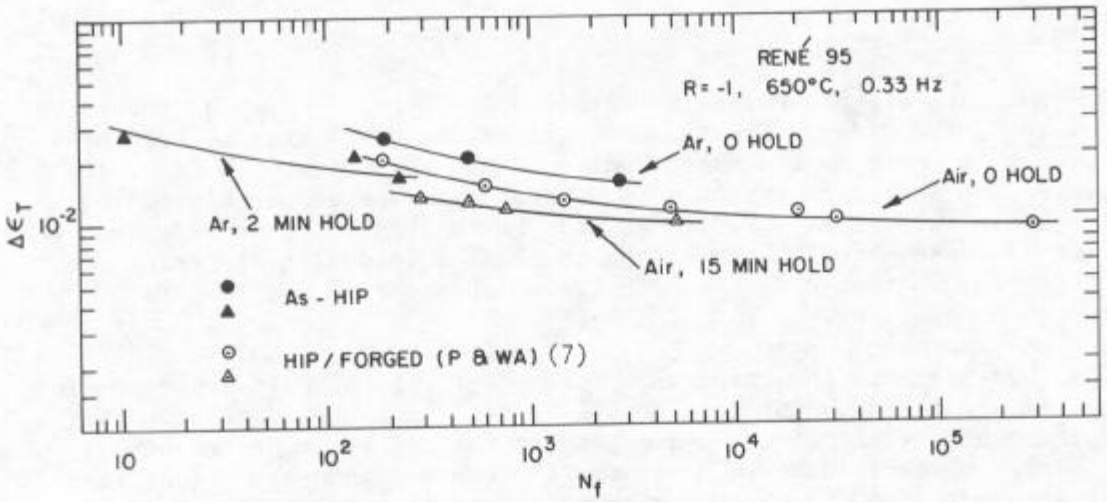
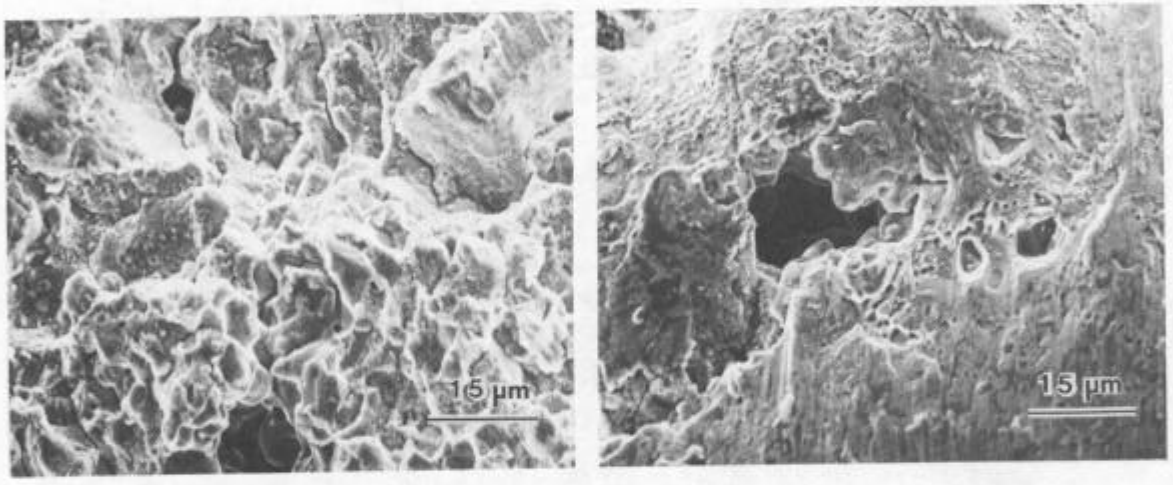
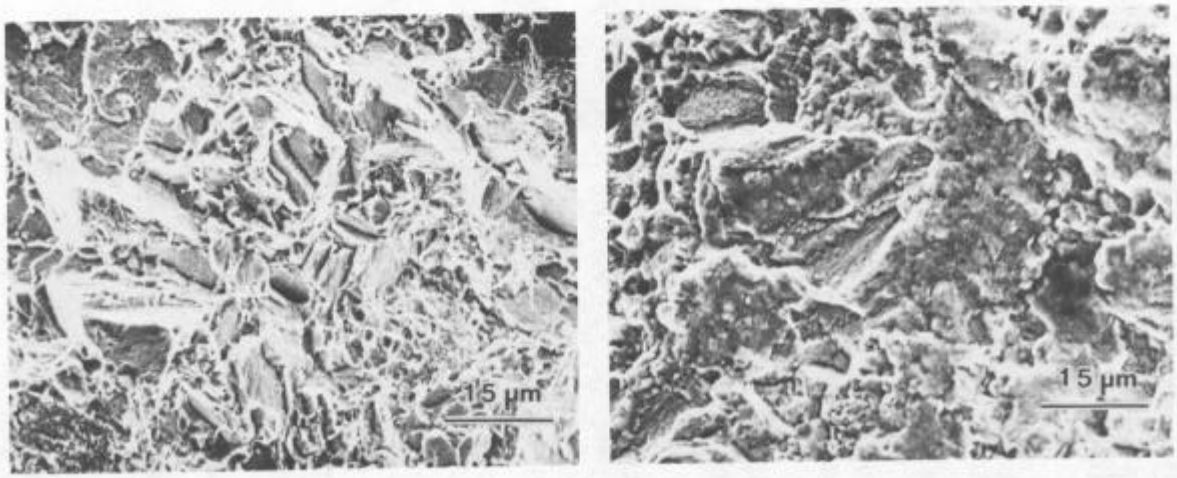


Figure 3 - Comparison between LCF behavior of as-HIP and HIP + forged materials.



a) b)

Figure 4 - LCF crack initiation associated with pores. a) 650°C, 0.33Hz, $\Delta\varepsilon_T$=2.7%, 120s hold b) 725°C, 0.33Hz, $\Delta\varepsilon_T$=1.52%



a) b)

Figure 5 - SEM fractographs showing a) faceted fracture surface at 650°C b) flat fracture at 725°C

B. FCG Behavior

The FCG data are presented in Fig. 6. A 2 min hold increases the crack growth rate by two orders of magnitude. It may be noted that a 6 min hold did not seem to cause more damage than the 2 min hold time. In fact, it seems to reduce the crack growth rate. These data are comparable to those found in other laboratories(7,8). It must be pointed out, however, that FCG rates for the same material may change at least 5 fold with different specimen geometries, so that comparisons between laboratories may lead to unwarranted conclusions.

Fig. 7 represents the comparison between growth rates in argon and in vacuum. If argon and vacuum were equally inert, one should observe the normal frequency effect (increased crack growth rate at lower frequency) due to time dependent damage. However, the reverse frequency effect seen here is attributed to the more aggressive nature of argon. The partial pressure of oxygen in argon was estimated to be about two orders of magnitude higher than that in vacuum.

Fig. 8 shows that crack propagation was TG with brittle striations under continuous cycling. The transition between room temperature continuous cycling and high temperature cycling with a hold time is shown in Fig. 9.

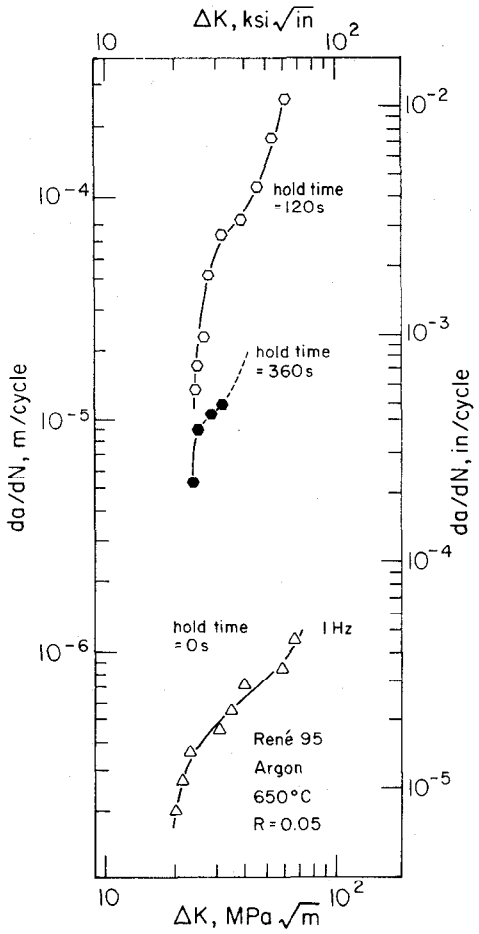


Figure 6 - Hold time effects on fatigue crack growth.

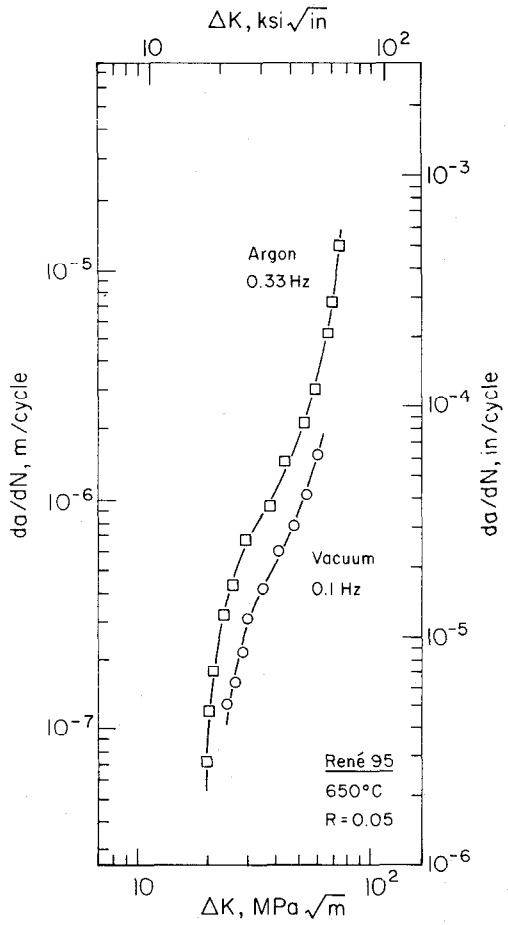


Figure 7 - Comparison between FCG results in argon and vacuum (5×10^{-5} torr on air).

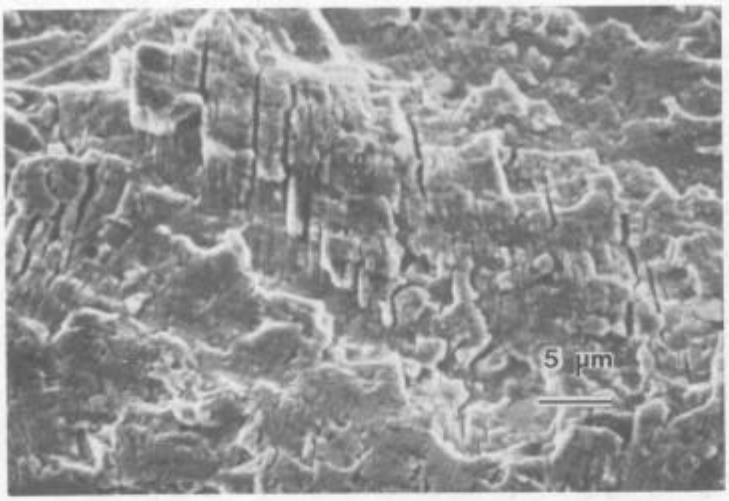


Figure 8 - TG cracking in a FCG specimen, 650°C, argon, 1Hz.

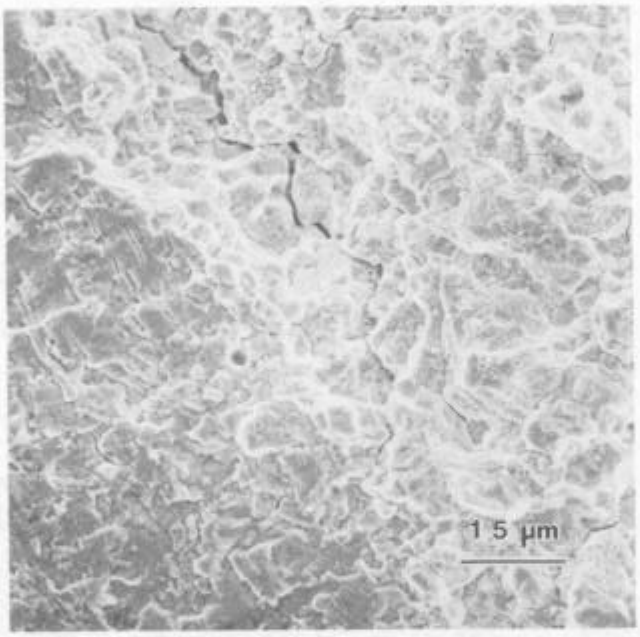


Figure 9 - Transition between room temperature TG cracking and high temperature IG cracking; 650°C, argon, 120s hold time.

High temperature crack growth was predominantly intergranular. A large number of secondary cracks was observed, usually along grain boundaries transverse to the stress axis. These were probably nucleated around the unsolutionized blocky γ' along the grain boundaries, see Fig. 10.

It may be pointed out here that no creep cavities were observed even with a 6 min hold time.

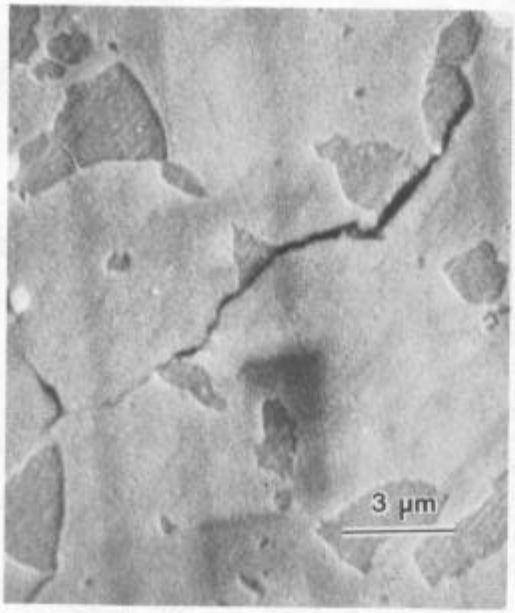


Figure 10 - Secondary cracking near coarse γ' at grain boundaries, 650°C, argon, 120s hold time.

Fig. 11 shows the depth profile of the dominant species on the fracture surface of the FCG specimen, continuously cycled in argon, as analysed by Auger electron microscopy. Since the chromium and oxygen profiles run parallel to each other up to 500 Å below the surface, it is believed that the fracture surface is covered by a chromium oxide layer of 500 Å depth: the oxide is probably Cr_2O_3. The oxide layers on the specimens tested with hold times were approximately 4-5 times thicker.

Discussion

Elevated temperature fatigue life is controlled by several competing factors. For example, creep processes at the crack tip may either enhance crack growth rates due to additional damage or retard FCG due to crack tip blunting. These processes are further complicated by simultaneous chemical reactions at the crack tip due to oxidation.

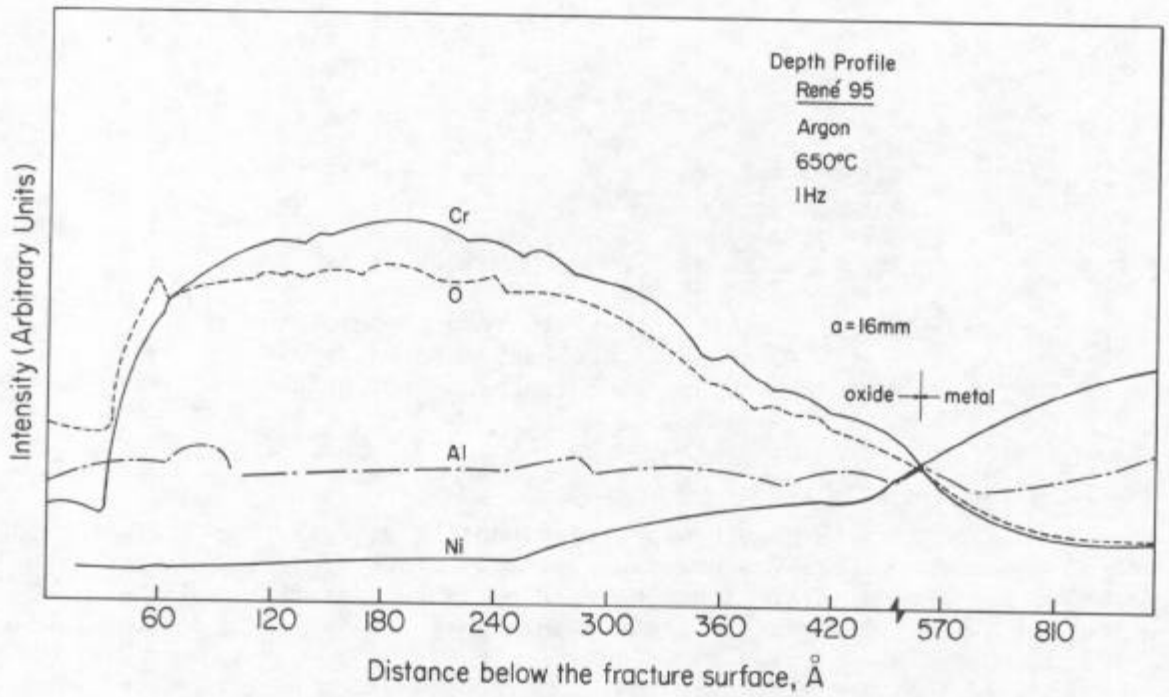


Figure 11 - Depth profile from the fracture surface by Auger Electron Spectroscopy.

The absence of creep cavities, lack of strong temperature dependence of LCF lives and the observation of comparable FCG rates for 2 min and 6 min hold times support the idea that the creep contributions to the FCG damage are relatively insignificant. It may be noted also that in a creep crack growth (CCG) test conducted in air, the CCG threshold K_{th} was found to be significantly higher (35MPa$\sqrt{m}$) than that in FCG (17MPa$\sqrt{m}$)(9). Basic thermodynamic calculations based on current creep cavity nucleation theories(10,11) indicate that if CO or CO_2 bubbles are formed, the bubble pressure would be less than 15Pa for our test conditions. This rules out the cavity nucleation process. The only possible contributions from creep processes could be local stress relaxation in strain controlled LCF tests and load controlled flow of the material ahead of the crack tip in FCG. Neither of these quantities can be measured easily and are insufficient to explain intergranular failure.

The constancy of the Coffin-Manson slope of the LCF curves (β=0.7), Fig. 2, suggests that there is no distinct change in fracture mechanism with hold times. Faceted fracture at 650°C, Fig. 5a), has been noted in other nickel base alloys(3, 12); it has been attributed to separation along intense planar slip bands. The disappearance of such facets at 725°C, Fig. 5b), may be due to more homogeneous slip occurring as temperature is increased.

Since all the fracture surfaces were covered with an oxide layer even in argon with 0.1-1ppm level oxygen, it is concluded that the time-dependent damage was predominantly environmental. In this respect comparison was made between oxidation of fresh fracture surfaces and oxidation of metallographically polished samples. It was noticed that the fresh fracture surface was more susceptible to oxidation. This indicates that static oxidation results and parabolic rate constants would be of no use in predicting the stress assisted crack tip oxidation kinetics. Therefore, it is believed that 2 min or 6 min hold times permit significant entrapment of oxygen at the grain boundaries. On subsequent cycling, cracking takes place along these weakened grain boundaries, resulting in the change in crack morphology from TG to IG or mixed mode. The increase in TG cracking rates may be explained on the basis of depleted chromium regions adjacent to the crack tip as a result of oxidation. Chromium acts as a solid solution strengthener and its depletion would probably reduce the local crack extension force. The volume change associated with oxide formation also should cause oxide-induced enhanced stresses at the crack tip. The idea of depleted species is not new; in aluminum alloys, reduced mechanical properties have been attributed to the formation of precipitate-free zones (13).

The oxidation occurring in high purity argon is indicative of very high reactivity of the fresh crack surfaces. This suggests that to minimize environmental effects, extremely low partial pressures of oxygen would be necessary and even purified argon is not a sufficiently inert medium for this type of testing.

Conclusions

1. LCF lives of René 95 were drastically reduced by 2 min hold times in argon at 650°C and 725°C.
2. FCG rates were two orders of magnitude higher with hold times of 2 min when compared with those of continuously cycled specimens.
3. Fracture paths changed from TG in the absence of hold times to predominantly IG with hold times.
4. Fracture surfaces in argon were covered with a chromium oxide layer of 500 Å thickness.

5. The time dependent-damage was found to be predominantly environmental.
6. High purity argon may not be an "inert" medium for this alloy.

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