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# THE EFFECT OF PRIOR TEMPERATURE CYCLING ON RUPTURE LIFE OF SUPERALLOYS

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## INTRODUCTION

The problem of life prediction of engineering components subjected to cyclic stresses and nonsteady temperatures is now receiving extensive attention [1]. Apart from concomitant and synergistic effects of creep, fatigue and environmental interaction, it is important to determine the effect of prior creep on fatigue failure and prior fatigue on creep rupture, i.e., a sequential exposure. In the case of prior fatigue, an influence on rupture strength may be due to propagation of fatigue cracks under static load, or may be due to a cyclic hardening or softening of the bulk material, i.e., a change in mechanical state.

A major source of cyclic strain may be the non-uniform expansion and contraction across a section of a component subjected to rapid temperature changes during operation. During start up and shut down of gas turbines, for example, temperature swings of the order 800 to 1000 K commonly occur and result in the generation of high strains in the thin edge portions of nozzle and bucket vanes [2]. Repetition of these transient strains may lead to initiation and propagation of cracks by thermal fatigue.

There is a second important source of cyclic strains associated with temperature changes which is not generally recognized. This is related to differences in coefficient of expansion between phases in many engineering alloys and can occur in a spatially uniform temperature field. It is this latter problem which is considered in the present paper.

Concern with the effect of temperature cycling on rupture strength of superalloys stemmed from observations of significant losses in high temperature strength of carbide fiber strengthened eutectic alloys after temperature cycling [3, 4, 5]. Since most superalloys contain carbides, it was reasoned that they too should be sensitive to this effect and experiments were performed to confirm this prediction [6, 7]. In general, it was found that the phenomenology was similar to that resulting from isothermal strain cycling. Thus, both cycling hardening and softening were observed, depending on the nature of the temperature cycle and the alloy properties.

Additional results on a nickel base superalloy, IN-738, and a cobalt base superalloy, MM509, are reported here. The treatment is mainly descriptive to emphasize the importance and generality of the problem. Some consideration is, however, given to the manner in which the temperature cycle controls cyclic strain accumulation.

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MS747

EXPERIMENTAL PROCEDURE

The compositions of the two alloys are given in Table 1. Both were produced in the form of cast slabs, 25 x 125 x 200 mm in section. The IN-738 was subsequently solution heat treated at 1303 K in hydrogen for 2 hours, cooled to room temperature and aged for 24 hours at 1113 K in argon. The MM509 was tested in the as-cast condition.

Temperature cycling was performed by induction heating and the temperature was controlled by a proportional band controller and a thermocouple attached to the specimen midsection. A digital waveform generator with hold time capability was used to control the set point on the controller and to produce the desired temperature cycle. Measuring thermocouples were located on the specimen gage section and close to the heads. By careful spacing of the coils, the top and bottom specimen heads were at a temperature between 0 and 15 K below the controlled portion of the gage length at the maximum cycle temperature. The temperature control was within ± 3 K. Additional details were described elsewhere [6].

Rupture specimens were centerless ground to final dimensions, with a 9.5 mm gage section of 2.54 mm diameter, prior to temperature cycling. Some tests on IN-738 were cycled in argon and tested in argon. The majority of the tests, however, involved both cycling and testing in air with $T_{max}$ = 1273 K and $T_{min}$ = 673 K. This cycle included a five minute hold at $T_{max}$ and $T_{min}$ and a heat up time of 2 minutes, 40 seconds giving a cycle period of 15 minutes and 20 seconds.

RESULTS

(i) Effect on Rupture Life

Figure 1 illustrates the effect of cycle period on rupture life of IN-738 at 1255 K and 151.7 MPa. For these experiments, the cycle was triangular and covered a temperature range between 1255 K and 700 K. Cycling and testing was in argon. For 50 cycles, a ten minute period caused an enhancement of life whereas a two hour period caused a loss of over 50%. A small number of very long cycles (period 40 hours) resulted in a smaller loss, whereas isothermal exposure for 100 hours at $T_{max}$ caused no life reduction. A final test with mixed cycles indicated that much of the beneficial effect of the fast cycling was retained even after slow cycling.

Figure 2 shows the effect of number of cycles between 1273 and 673 K with a five minute hold for air exposure and testing. The rupture test temperature of 1144 K was chosen since previous work on nickel base superalloys has demonstrated that high temperature isothermal exposure in air can lead to a drastic reduction in tensile ductility with a maximum effect in this temperature range [8, 9]. Rupture life was severely reduced after 560 cycles. An appreciable loss in life was also observed for isothermal exposure with a total time at $T_{max}$ greater than the accumulated time at $T_{max}$ in the cycled specimens. Since isothermal exposure at a similar temperature in argon did not cause any reduction in life (Figure 1), it appears likely that this is associated with localized oxidation effects as described by Chang [8] and Wood [9]. However, equivalent degradation is achieved after 100 cycles involving an accumulated time at $T_{max}$ of only 8 hours. Apart from a possible slight increase in rupture strength after 25 cycles, the trend is for a progressive degradation with increasing number of cycles for these cycle conditions.





The MM509 data shown in Figure 3 also demonstrate a dramatic loss in rupture life with increasing number of cycles for tests at 1144 K and 137.9 MPa. After 560 cycles, the rupture life was reduced by about 87%.

For this cobalt-base alloy, the isothermally exposed specimens showed as large a decrease in rupture life as the cycled specimens. Since inert atmosphere tests were not performed, it is not clear whether the effect is related to precipitate changes or to environmental effects. Ductility was not measurably influenced by any of the exposures in this alloy or in the IN-738.

(ii) Microstructural Studies

One specimen of both alloys for each exposure condition was mounted longitudinally and examined optically. Both alloys failed intergranularly for all initial exposures (or possibly interdendritically for the MM509). No clear differences were observed between specimens. Typical microstructures (Figure 4) demonstrate the major structural differences in the two alloys. In the IN-738, the MC carbide is estimated to occupy about 1 vol. %, whereas in the MM509 about 8 vol. % total carbide consists of approximately equal amounts of MC and $M_{23}C_6$.

Transmission microscopy was performed on specimens uncycled, isothermally exposed, and cycled 560 times for both alloys. The samples were taken prior to rupture testing. There did appear to be a higher dislocation density in the cycled IN-738 although the sample was too small to be certain. There were no detectable differences between the three MM509 specimens. Nevertheless, there were clear indications of deformation at precipitate/matrix interfaces in all samples.

Figure 5 shows a heavy concentration of dislocations in the matrix at a precipitate interface in the cycled IN-738. The darker contrast in the matrix in this region is probably the result of an in situ reaction of the form MC + $\gamma \rightarrow M_{23}C_6 + \gamma'$. Figure 6, which is from an uncycled specimen, confirms the development of plastic deformation at the interface during cooling and also shows similar dislocation generation from an unidentified particle trapped within the carbide. This effect was quite common. Figure 7 shows an area in the cycled MM509 with clear alignment of the $M_{23}C_6$ carbide faces and slip planes and extensive stacking fault formation. However, as previously mentioned, all exposures gave similar structures in the areas examined.

Uncycled samples were subjected to temperature cycling between room temperature and about 1073 K using the hot stage of a JEOL 200 KV microscope. The cycle period was about 5 minutes. The IN-738 sample became contaminated quite readily so that clear observation was difficult. However, MM509 was more resistant and several cycles were completed with photographs being taken at room temperature after each cycle. A precipitate interface dislocation structure is shown in Figure 8 after 0, 2 and 4 cycles. It was observed that the dislocations moved out of the field during heating and new dislocations were nucleated at the interface during the cooling cycle. The temperature range for dislocation motion on heating and nucleation on cooling was in both cases between 673 and 873 K. This motion away from the precipitates probably accounts for the similar microstructures observed in bulk cycled specimens. Nevertheless, cyclic straining is occurring as a result of the expansion coefficient mismatch (of the order $10^{-5}$ $K^{-1}$ [5]) and the detailed dislocation loop complex is different after each cycle.

## DISCUSSION

There are at least three factors involved in influencing the observed major effects of cyclic temperatures on rupture life. Clearly, changes in the amount, distribution or type of precipitate normally associated with long term isothermal exposure of superalloys will also occur during temperature cycling and may significantly change the mechanical properties. For IN-738, since no change in rupture life was observed for argon exposed specimens, this factor is not considered important for the conditions examined. It may well be very significant for MM509 since isothermal exposure of this alloy for 100 hours at 1273 K has previously been shown to reduce the rupture life by about 85% [10]. This loss was attributed to the combined effects of carbide agglomeration plus partial breakdown of MC and reprecipitation as acicular $M_{23}C_6$. However, it is possible that a second important factor here is the grain boundary weakening due to oxygen diffusion. Although the most dramatic manifestation of this phenomenon has been in the tensile ductility loss, which could be restored by removing a surface layer to the depth of grain boundary penetration, [8, 9] there is some indication of a damaging effect on rupture life also [9]. This effect could, therefore, account for much of the loss in life of the isothermally exposed IN-738. There have been no systematic studies of this effect in cobalt base alloys but it may well be an important contributing factor here too.

The third important factor, which was introduced in the beginning of this paper, is the cyclic strain (and associated hardening and softening) induced as a result of the differences in coefficient of expansion between phases in the alloys. During cooling from some initial stress free state, the stress development around a dispersed phase resembles that in shrink fitting if the matrix has a larger thermal expansion coefficient. Laslo [11] developed the stress analysis techniques for a variety of such stresses as they might occur in different ideal microstructures, labelling them "tessellated stresses". For uniformly dispersed spheroids, the stress analysis of the matrix becomes that of the pressure on the inner wall of a thick spherical shell, producing radial compression and tangential tension. Yielding of the matrix may readily occur, particularly if the particles are not spherical [12].

The effect of cyclic heating and cooling on the development of precipitate induced tessellated stresses has apparently not been considered, but it is clear that if local plastic deformation occurs in tension on cooling, reheating to the starting temperature will produce residual compression. Plastic deformation in compression may occur during the heating period or during hold at maximum temperature. Clearly, the heating and cooling time, hold time, $\Delta T$, and $T_{max}$ all affect the shape of the local hysteresis loop including the stress range and plastic strain range. The in situ electron microscope study of MM509 demonstrated plastic deformation during both the heating and cooling cycle for the particular cycle studied and confirmed that local cyclic straining following thermal excursions can lead to significant microstructural changes.

## CONCLUSIONS

1. There are at least three important phenomena associated with temperature cycling of multiphase alloys: (i) phase instability, (ii) environmental effects and (iii) local cyclic straining resulting from tessellated stresses. All may have an important effect on mechanical properties.





2. Cyclic temperature excursions may lead to appreciable changes in rupture life in nickel and cobalt base alloys. The magnitude of these changes is sensitive to the nature of the temperature cycle and the number of cycles.

3. Electron microscopy confirmed the local deformation in the matrix surrounding precipitates and, by heating and cooling a specimen in the microscope, demonstrated cyclic strain accumulation.

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Table 1 Compositions of Alloys (wt %)

| Alloy | Ni | Co | Cr | Al | Ta + Cb | | C | W | Fe | Mo | Ti | Zr | B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| IN-738 | Bal | 8.42 | 15.34 | 3.38 | 1.78 | 0.86 | 0.1 | 2.6 | 0.14 | 1.82 | 3.54 | 0.05 | 0.011 |
| MM509 | 10 | Bal | 23 | -- | (3.59) | | 0.63 | 7 | <0.1 | <0.1 | 0.19 | 0.47 | <0.01 |

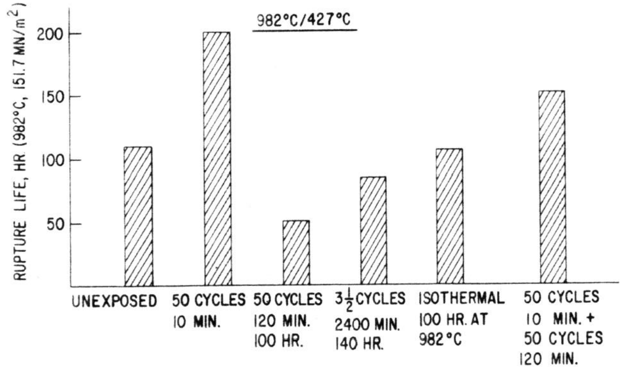


Figure 1 Effect of Prior Temperature Cycling on Rupture Life in IN-738 at 1255 K and 151.7 MPa. Triangular Cycle Between 700 K and 1255 K. Exposure and Testing in Argon

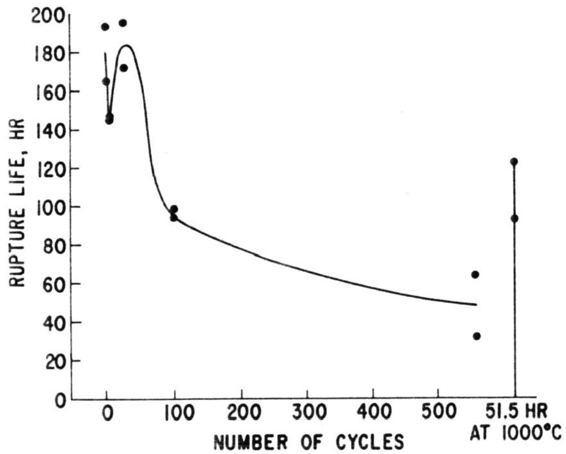


Figure 2 Effect of Number of Temperature Cycles and Isothermal Exposure on Rupture Life of IN-738 at 1144 K and 275.8 MPa. Cycling Between 673 K and 1273 K with 5 minute hold. Exposure and Testing in Air





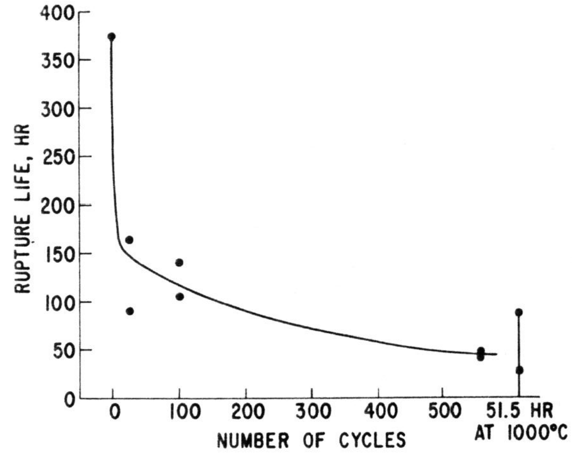


Figure 3 Effect of Number of Temperature Cycles and Isothermal Exposure on Rupture Life of MM-509 at 1144 K and 137.9 MPa. Cycling Between 673 K and 1273 K with 5 minute hold. Exposure and Testing in Air

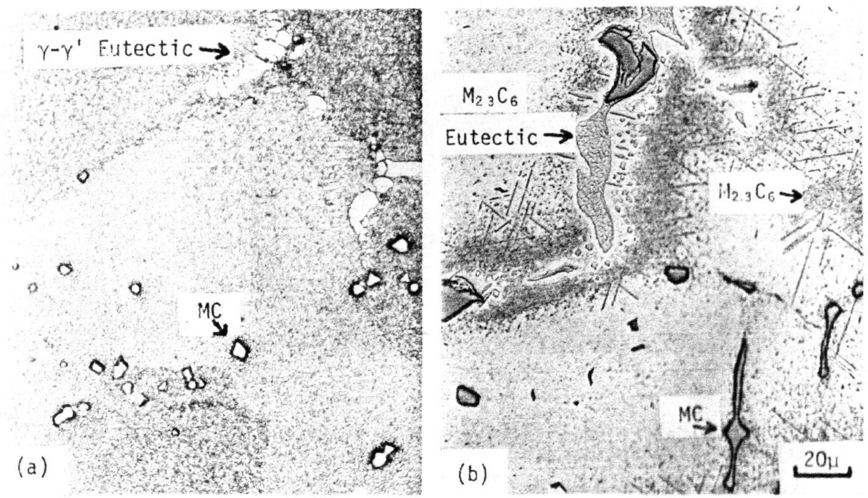


Figure 4 Typical Microstructures Showing Major Phases in (a) IN-738 and (b) MM-509

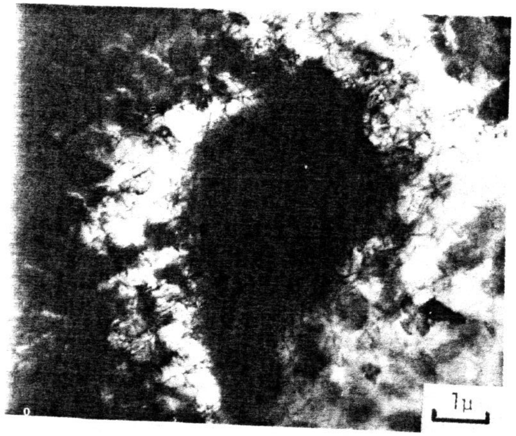


Figure 5 IN-738 Cycled 560 Times Showing Heavy Concentration of Dislocations in Matrix Adjacent to Precipitate

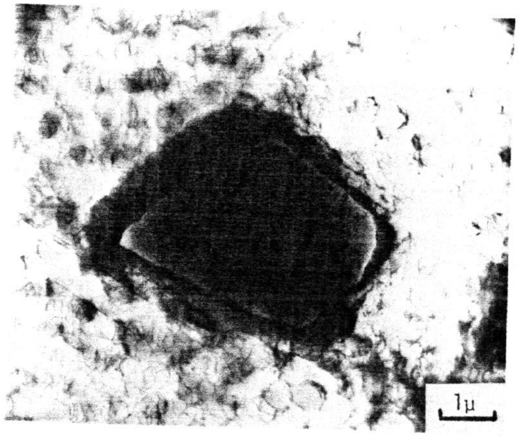


Figure 6 Isothermally Exposed IN-738 Showing Dislocation Generation Both Within Carbide and at the Matrix Interface





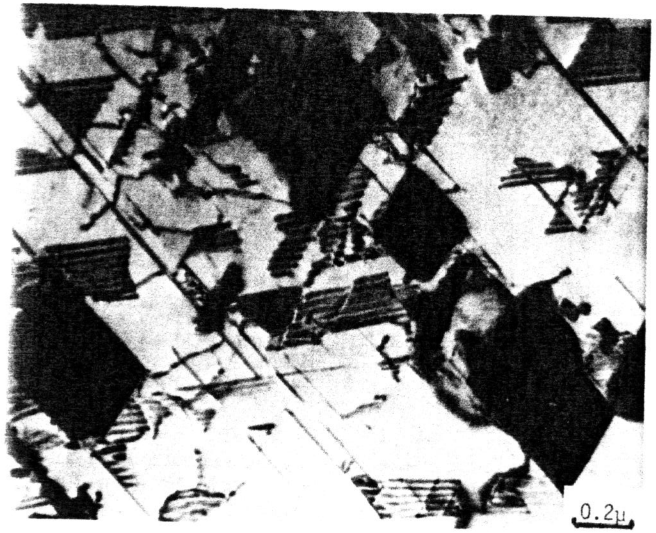


Figure 7 MM-509 Cycled 560 Times Showing $M_{23}C_6$ Carbides and Associated Slip


Figure 8 Dislocation Generation at Carbide Interface in MM509 Resulting from Temperature Cycling in the Electron Microscope
(a) Initial Structure

continued

continued

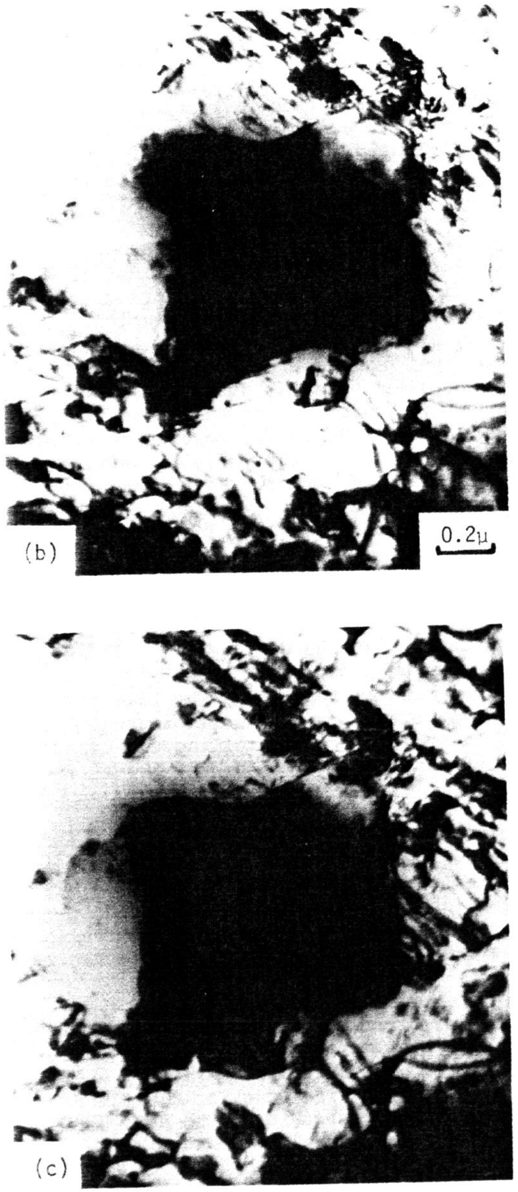


Figure 8 Dislocation Generation at Carbide Interface in MM509 Resulting from Temperature Cycling in the Electron Microscope
(b) After 2 Cycles
(c) After 4 Cycles