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The effect of frequency on the rate of fatigue crack propagation in MAR M002 single crystals has been studied at a test temperature of 600°C. Selected area channeling patterns have been used to measure the crack-tip plastic zone size, and the strain gradient within these zones.

At 600°C, at similar  $\Delta K$ , the plastic zone size increases as the frequency is reduced in a way consistent with the expected plastic relaxation. At this  $\Delta K$ , the crack tip strain was frequency independent at 600°C, and of twice the magnitude of that observed in room temperature tests. This is discussed in terms of the increasing homogeneity of slip with increasing temperature.

#### INTRODUCTION

During fatigue at high temperature, it is to be expected that creep deformation will become increasingly important as the test frequency is lowered. In polycrystalline materials both grain boundary sliding and cavity nucleation at grain boundary precipitates are of importance in this context. In the present work a single crystal nickel based superalloy has been chosen (MAR M 002) in order to eliminate grain boundary effects, and the object of the work has been to investigate the deformation processes at the tip of fatigue cracks propagated at elevated temperatures in this material, and their variation with test frequency.

#### EXPERIMENTAL METHODS

Single crystal blocks of unidirectionally solidified (UDS) MAR M002 were available, with a growth direction of [001]. The composition of the material was as given in Table 1.

Table 1: Composition of the UDS MAR M002 (wt %)  
Co 10; Cr 8.8; Al 5.5; Ti 1.4; W 9.8; Ta 2.5;  
Hf 1.5; C 0.15; B 0.012; Zr 0.07; Ni balance

The crystals were heated for 1 hr at 1100°C followed by 16 hr at 860°C. Their microstructure consisted of approximately 60 volume percent of cuboidal  $\gamma'$  of 0.5 $\mu\text{m}$  edge dimension, together with about 6 volume percent of a  $\gamma/\gamma'$  eutectic. A number of interdendritically located 'script' carbide particles were present.

Centre-cracked tension (CCT) specimens were spark machined from the blocks such that each of the faces was close to {100}. The material was then ground to the appropriate specimen dimensions (30mm x 2mm x 105mm).

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Fatigue tests were conducted in air at room temperature and at 600°C, using a servo-hydraulic testing machine operating under conditions of constant  $\Delta K$  at an R value of 0.1. Triangular loading at test frequencies of 10 Hz, 1 Hz, 0.1 Hz was applied. Crack extension was monitored using a DC potential drop method. This enabled absolute values of crack length to be determined to within 0.05 mm.

The local strain distribution around the tip of the fatigue crack was examined in specimens from interrupted tests, subject to a  $\Delta K$  of 23.4 MPa m<sup>1/2</sup> at each of the three test frequencies at 600°C, together with a further specimen tested at the same  $\Delta K$  at room temperature at 10 Hz. In each case mid-thickness sections through the crack tip were prepared, and Selected Area Channelling Patterns (SACPs) observed using a JEM 100C microscope with TEMSCAN attachment.

The position of the elastic/plastic interface was determined by the systematic interrogation of the SACP quality obtained from small volumes of material having a surface diameter of 2 - 3µm. Since a departure from lattice perfection also results in a quantifiable deterioration in the quality of the SACP (1), the use of a calibration from a series of specimens deformed to known strains permitted the measurement of the strain distribution within the plastic zone.

EXPERIMENTAL RESULTS

In fig.1, a family of da/dN versus  $\Delta K$  curves is presented for a test temperature of 600°C, obtained at frequencies of 10 Hz, 1 Hz & 0.1 Hz. There is seen to be an increase in  $K_c$  as the frequency decreases, and also an increase in the threshold  $\Delta K$  value, and these effects have been discussed elsewhere (2). At intermediate values of  $\Delta K$  the curves of fig.1 are seen to cross, and the variation of growth rate with frequency is most readily appreciated from fig.2, where da/dN versus test frequency is plotted for a  $\Delta K$  value of 23.4 MPa m<sup>1/2</sup>. There is seen to be a decrease in da/dN as the frequency is increased from 0.1 Hz to 1 Hz, and a slight increase in da/dN as the frequency is changed from 1 Hz to 10 Hz.

The Crack Tip Plastic Zone

Zone sizes. The crack tip plastic zones obtained from the SACP technique are shown in fig.3. It may be seen that there exists only a relatively small difference between the plastic zone sizes of the specimens deformed at room temperature and that deformed at 600°C and 10 Hz. Values of the plastic zone sizes measured directly ahead of the crack tip,  $r_p$ , are presented in Table 2.

TABLE 2 - Plastic Zone Size Measurements obtained by the SACP Technique

Stress Intensity	$\Delta K = 21.6 \text{ MPa m}^{1/2}$	$\Delta K = 23.4 \text{ MPa m}^{1/2}$			$\Delta K = 45 \text{ MPa m}^{1/2}$
Test temp.	Room Temperature	600°C	600°C	600°C	600°C
Frequency	10 Hz	10 Hz	1 Hz	0.1 Hz	0.1 Hz
$r_p$ (µm)	158	178	223	656	1210

As the frequency is lowered at 600°C, a progressive increase in  $r_p$  is observed. Although the increase is only small for the 1 Hz test, the 0.1 Hz specimen has a value of  $r_p$  over four times greater than that observed in the room temperature test.

In order to demonstrate the effect of the applied stress intensity upon the plastic zone size at 600°C and 0.1 Hz, a further plastic zone was measured when  $\Delta K$  was 45 MPa m<sup>1/2</sup>. The result of this, together with that for a  $\Delta K$  of 23.4 MPa m<sup>1/2</sup> is shown in fig.4.

Strain distribution within the plastic zones. The variation of plastic strain with distance directly ahead of the crack tip,  $d_{pz}$ , was measured for each of the specimens represented in fig.3. The distribution of plastic strain as a function of  $d_{pz}$  is shown in fig.5. Each point represents the measured strain associated with a small volume of material with a surface area 2 - 3µm in diameter. The elastic/plastic interface determined previously can be seen to correspond to a plastic strain of approximately 1%.

It is evident from fig 5 that the specimens deformed at 600°C tend towards the same high local strain at the crack tip. However, as the frequency is lowered there is not only an increase in the plastic zone size, but also a progressive decrease in the strain gradient. The room temperature specimen appears to exhibit a similar strain gradient to that of the 600°C, 10 Hz specimen, but it shows a significantly lower maximum strain at the crack tip.

#### Metallography

As reported elsewhere (2), at room temperature the cracks propagate parallel to both {100} and {111}, but at 600°C there is less evidence of the latter path. The relatively large areas of growth parallel to {100} show large numbers of 'script' carbide particles. The SEM micrographs of fig.6 illustrate features apparent in the specimens tested at 0.1 Hz. Fig.6a shows a series of regular perturbations upon the crack surface: their spacing (~0.5µm) is close to the crack growth rate observed at the operating  $\Delta K$ . Fig. 6b shows the prolific cracking of the script carbides that is observed within the plastic zone ahead of the crack tip at high values of  $\Delta K$ . Although some cracking of carbides is observed at the higher frequencies of testing, it is usually limited to small areas directly ahead of the crack tip. At high values of  $\Delta K$  in the 0.1 Hz test, however, cracking of carbides both ahead and to the side of the crack propagation plane is found.

#### DISCUSSION

##### Plastic Zone Sizes

The SACP method used here has the advantage of being able to measure plane strain plastic zones. The magnitudes of  $r_p$  recorded in fig.3, in comparison with crack length and specimen size indicate that the test-piece may be considered to be in a condition approaching plane strain at its central cross-section.

It is clear from Table 2 that the values of  $r_p$  observed in the room temperature test and the 10 Hz 600°C test are similar. In general the relationship between the applied stress intensity factor and the monotonic plastic zone size  $r_p$  can be expressed (3) as:

$$r_p = \alpha(K/\sigma_y)^2 \quad (1)$$

where  $\alpha$  is a dimensionless factor and  $\sigma_y$  the yield stress. Using this relationship it is possible to show that the room temperature data would predict, for the  $\Delta K$  operating at 600°C, a value of  $r_p$  of approximately 170µm. This value represents the situation in which both temperature and frequency have little effect, and is close to the observed value of 178µm for the

10 Hz test at 600°C. If, however, the predicted value is compared to the observed values at 600°C at 1 Hz and 0.1 Hz, an increasing discrepancy is apparent. During each cycle at 1 Hz it would appear that relaxation has occurred, with the result that the value of  $r_p$  is greater than that observed at 10 Hz by 45µm. A further tenfold decrease in the test frequency is associated with a further relaxation, resulting again in a plastic zone size which is larger than predicted. Comparison of the values of  $r_p$  at 10, 1 and 0.1 Hz can be obtained by considering the value of the ratio:

$$\frac{r_p(0.1 \text{ Hz}) - r_p(10 \text{ Hz})}{r_p(1 \text{ Hz}) - r_p(10 \text{ Hz})}$$

For the measured values of  $r_p$ , this ratio is 10.6, which is in reasonable agreement with a simple time-dependent relaxation process, which would yield a value of 10. Hence it would appear that the observed increase in the plastic zone size is due to an increasing creep relaxation of the crack tip stresses. However, the effect of increasing  $\Delta K$  upon the relaxed plastic zone size is considerably less than would be predicted.

If no relaxation occurs at the crack tip, a value of  $r_p$  of 630µm would be expected. However, extrapolation of the data presented in Fig. 3 in which creep relaxation has occurred, predicts a value of  $r_p$  of 2430µm at  $\Delta K = 45.0 \text{ MPa m}^{1/2}$ , whereas the observed value is 1210µm. This disparity is to be expected when the nature of the relaxation processes are considered. An increasing plastic zone size will be one relaxation process, but relaxation may arise from the formation of cracks at local inhomogeneities (4) such as the script carbides (Fig. 6b). Relaxation by secondary cracking will become more predominant as the applied stress intensity range rises, so that there will be a smaller increase in the size of the plastic zone by creep relaxation.

#### Strain Distribution Within the Plastic Zone

It is apparent from Fig. 5 that the crack tip plastic strain of ~14% in the specimens tested at 600°C appears to be independent of frequency. This suggests that no essential change in the crack tip deformation mechanism occurs as the test frequency is lowered. The results from the specimen tested at room temperature show a crack tip strain of ~8% (for a similar applied  $\Delta K$ ), and this temperature decrease is found to cause a two-fold decrease in  $da/dN$  at this  $\Delta K$  (2). A change in mechanism with change of temperature is suggested by these observations, coupled with the fact that a decreased amount of {111} fracture facets are found at the higher temperature. Recent work (5,6) has also reported extensive {100} crack extension at elevated temperatures in similar material.

Since the strain at the crack tip can be considered to be directly related to the crack tip opening displacement (CTOD), it is evident that this change in crack propagation mechanism is associated with an increase in the CTOD. It has been suggested (7) that oxidation at the crack tip will reduce the degree of slip reversibility, thus giving an increased CTOD and an increased  $da/dN$ . However, the crack tip strains for the 10, 1 and 0.1 Hz tests are approximately the same, despite the 100-fold difference in the time of exposure of the newly created crack surface to an oxidizing atmosphere. In the present case, therefore it is thus improbable that oxidation causes the change in mechanism with increasing test temperature. An alternative approach is to consider the change in slip geometry with temperature.

Other workers (8, 9) have shown in a similar material that, as the temperature increases, the tendency for intense planar slip is reduced. At

room temperature, crack growth will be dominated by a single slip plane, giving rise to {111} fracture facets. This will result in a tendency for crack extension by a process similar to Mode II, and consequently there will be a relatively small CTOD associated with it. At 600°C, a greater number of slip systems will become active and crack growth will no longer be dominated by single slip. The more homogeneous deformation around the crack tip will lead to the development of a more symmetrical plastic zone. Hence growth parallel to {100} will be observed, resulting in the larger CTOD associated with Mode I type processes.

SUMMARY AND CONCLUSIONS

From da/dN vs.  $\Delta K$  curves determined at 600°C there is seen to be an increase in  $K_C$  and in the threshold  $\Delta K$  as the frequency decreases. At intermediate  $\Delta K$  values (e.g. 23.4 MPa m<sup>1/2</sup>) a marked increase in da/dN as the frequency is decreased from 1 Hz to 0.1 Hz, is also observed.

The plane strain plastic zone sizes at the crack tip have been measured by the SACP technique. At 600°C, with  $\Delta K = 23.4$  MPa m<sup>1/2</sup>, the plastic zone size increases as the frequency is reduced. It has been shown that this increase is consistent with the expected plastic relaxation during the stress cycle. At the lowest frequency of testing, 0.1 Hz, fracture surface perturbations were observed whose spacing was comparable with (the increased) da/dN.

At high  $\Delta K$  values, the increase in  $r_p$  is less than expected by extrapolation of the lower  $\Delta K$  measurements. This arises because relaxation by the formation of subsidiary cracks at the 'script' carbides takes place, thus reducing the amount of direct creep relaxation required.

Strain distribution within the plastic zone has been measured, and it is found that the crack tip strain is frequency-independent at 600°C, and of twice the magnitude of that observed in room temperature tests. A decreased amount of {111} fracture facets are formed at the higher temperature, and these are associated with crack growth dominated by a single slip plane and a correspondingly small CTOD. The principal effect of the temperature increase is to promote slip activity on many planes at the crack tip, leading to a Mode I-type of growth parallel to {100}. This is associated with an increased CTOD and thus higher crack tip strain than the room temperature mechanism.

ACKNOWLEDGEMENTS

The authors are grateful to Professor Sir Peter Hirsch, FRS, for laboratory facilities made available, and also to the SERC and to the Procurement Executive of the Ministry of Defence for support.

SYMBOLS USED

- $d_{pz}$  = distance ahead of crack tip within the plastic zone ( $\mu\text{m}$ )
- da/dN = crack growth rate per cycle (mm/cycle)
- $K_C$  = fracture toughness (MPa m<sup>1/2</sup>)
- $K_{max}$  = maximum stress intensity factor (MPa m<sup>1/2</sup>)
- $K_{min}$  = minimum stress intensity factor (MPa m<sup>1/2</sup>)
- $\Delta K$  = stress intensity range (MPa m<sup>1/2</sup>)

- $r_p$  = plastic zone size directly ahead of the crack tip ( $\mu\text{m}$ )
- $R = K_{\min}/K_{\max}$
- $\alpha$  = constant relating  $r_p$  and  $(K/\sigma_y)^2$
- $\nu$  = frequency of testing (Hz)

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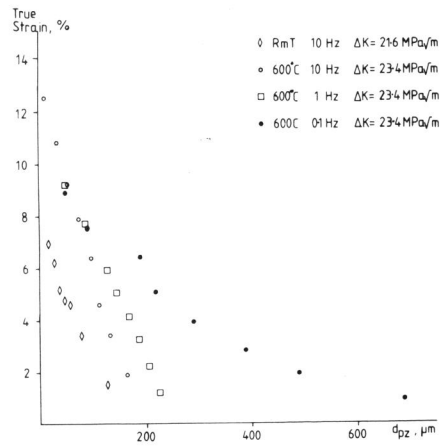


Fig.5. Strain distribution ahead of crack tip.

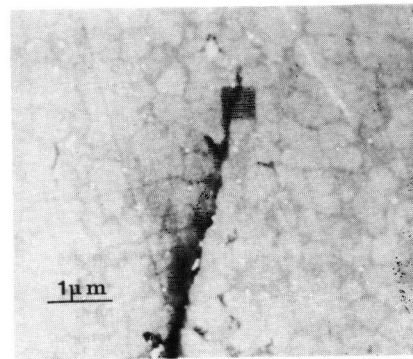


Fig.6a. T = 600°C, frequency = 0.1 Hz,  $\Delta K = 23.4 \text{ MPa}\sqrt{\text{m}}$

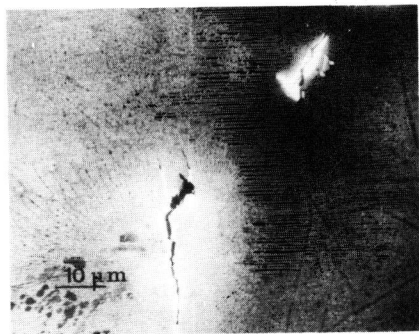


Fig.6b. T = 600°C, frequency = 0.1 Hz,  $\Delta K = 45 \text{ MPa}\sqrt{\text{m}}$

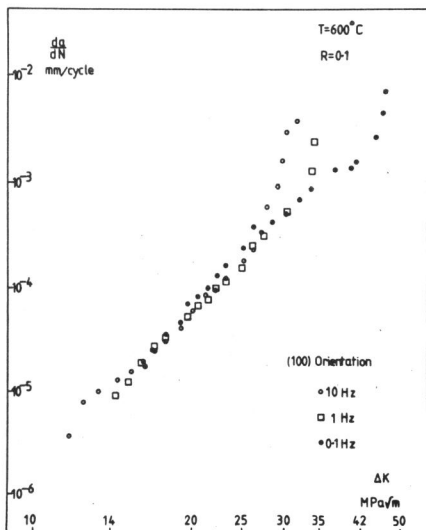


Fig. 1.  $da/dN$  vs.  $\Delta K$  at 600°C.

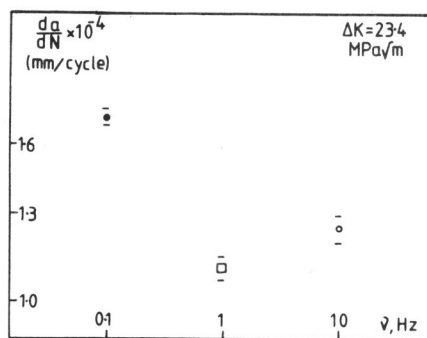


Fig. 2.  $da/dN$  vs.  $\nu$  at  $\Delta K = 23.4 \text{ MPa m}^{1/2}$ .

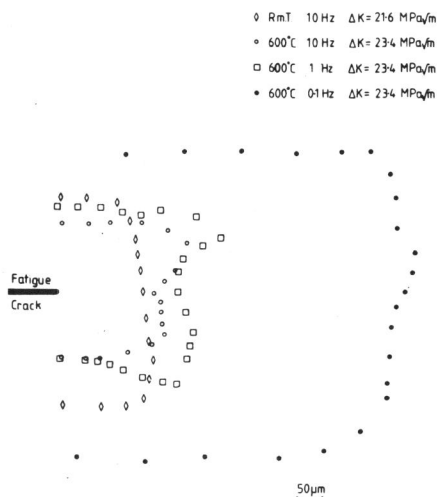


Fig. 3. Effect of frequency and temperature on plastic zone size.

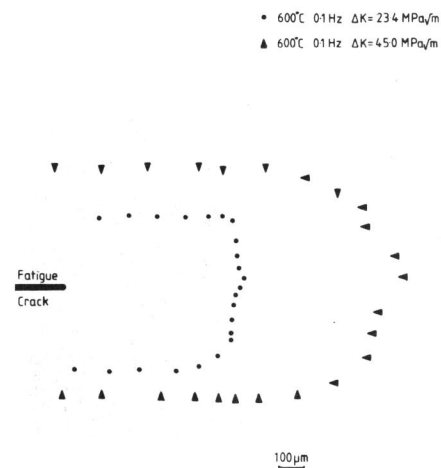


Fig. 4. Effect of  $\Delta K$  on plastic zone size.