

# FATIGUE OF AN ALUMINIDE COATED NICKEL-BASE SUPERALLOY AT 600°C – EFFECTS OF BRITTLE PRE-CRACKING

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## ABSTRACT

The fatigue behaviour of an aluminide coated single crystal nickel-base superalloy has been studied at 600°C. Fully-reversed uniaxial tension fatigue tests were performed on (100)-oriented hollow cylindrical specimens in extension control. Total strain ranges from 0.5% to 1.4% were investigated; specimens were tested both in the as-received condition (following coating and substrate heat treatment) and after brittle monotonic precracking of the coating at room temperature. Narrow and wide precracks were generated. Coating cracking in the first fatigue cycle reduced fatigue lives above a total strain range of 0.7% for non-precracked specimens. No crack growth from narrow room temperature precracks was observed at any of the strain ranges tested. However, crack growth was observed in specimens with wide precracks. The results are interpreted in terms of oxidation induced crack closure and the fatigue threshold values for the substrate alloy.

## KEYWORDS

Fatigue, nickel-base alloys, aluminide coatings, single crystals, elevated temperature, crack closure.

## INTRODUCTION

The development of high-strength nickel-base superalloys for use in gas turbine blade applications has led to a general decrease in their corrosion resistance. High-temperature aluminide coatings were developed in the 1960's to provide adequate protection against environmental degradation of high strength blade alloys (Meetham, 1986). Aluminide coatings are formed by a diffusion reaction of an aluminium source with a nickel-base alloy substrate to produce a (typically) 50-75  $\mu\text{m}$  layer of  $\beta\text{-NiAl}$  which serves as a reservoir of aluminium to form the stable protective oxide  $\text{Al}_2\text{O}_3$  (Goward and Boone, 1971).

However, the coating is brittle at lower temperatures (below  $\sim 750^\circ\text{C}$ ) due to the inherent brittleness of NiAl (Ball and Smallman, 1966). Brittle cracking of the coating can lead to reduced fatigue lives in coated components (Grünling et al., 1987). Numerous studies have

been performed to characterise the extent to which the presence of the coating effects the mechanical behaviour of the superalloy substrate; many of these are summarized in a review performed recently by Wood (1989). Varied results have been obtained. Some researchers have reported decreased fatigue lives while others have observed no effect. The way in which the presence of the coating affects the behaviour of the substrate appears to be a complex function of many factors, including cycle frequency, magnitude of applied stresses, test temperature and coating parameters such as thickness and aluminium content.

This paper presents the results of a study on the mechanisms of fatigue crack initiation and growth in an aluminide coated single crystal nickel-base superalloy at 600°C. At this temperature the coating behaves in a brittle manner, but oxidation and other temperature effects are also expected to contribute to the overall behaviour. Tests were performed on as-received specimens which had been coated and given substrate heat treatments, and specimens following brittle precracking of the coating at room temperature. The effects of precracking were investigated both as a means of further examining the mechanisms of fatigue crack initiation and growth and as a simulation of possible cracking of the coating during blade handling or due to the application of high strains at low temperatures.

## EXPERIMENTAL PROCEDURES

Solution heat treated single crystal bars of a nickel-base superalloy, with axial orientations within  $\pm 5^\circ$  of a  $\langle 100 \rangle$  direction, were machined into hollow cylindrical specimens (outside diameter 6.4 mm, wall thickness 1.0 mm, and gauge length 15 mm). A high-activity aluminide coating approximately 50  $\mu\text{m}$  in thickness was formed by aluminisation at 870°C followed by a one hour diffusion treatment at 1100°C. A final ageing step of 16 hours at 870°C produced a bimodal distribution of  $\gamma$  in the substrate. The microstructure of the coating following the ageing treatment consisted of a main coating region of  $\beta$ -NiAl with fine  $\text{M}_{23}\text{C}_6$  and  $\gamma$  precipitates and a transition zone of elongated  $\text{M}_{23}\text{C}_6$  precipitates in a  $\beta$  phase matrix (the coating microstructure can be seen in Figure 5).

The fatigue behaviour of as-aged specimens was investigated at 600°C over total strain ranges from 0.5% to 1.4%. Fully-reversed ( $R = -1$ ) fatigue tests were performed in extension control. A triangular waveform was employed to give a constant strain rate of  $3.5 \times 10^{-3} \text{ sec}^{-1}$ . Tests at 600°C were also performed on specimens in which the coating had been precracked at room temperature. Two widths of precrack were examined: narrow precracks formed by monotonic loading to just beyond the coating fracture strain ( $\sim 0.45\%$  at room temperature), and wide precracks formed by loading to a substantial plastic strain ( $\sim 2.0\%$ ). The testing matrix and fatigue results are shown in Table 1.

Fatigue life data for all tests were recorded as cycles to failure. The initial load response was noted, but hysteresis loops were not recorded as the applied stresses were fully elastic for all strain ranges investigated. Analysis of failed specimens was carried out using light microscopy and scanning electron microscopy (SEM). Fracture surfaces were first observed using a low-magnification stereo microscope and then using a SEM. Metallographic cross-sections were prepared and examined using standard techniques.

## RESULTS

**As-Aged Specimens.** The fatigue data for as-aged (non-precracked) specimens are shown in Table 1 and Figure 1. All of the data points fall into one scatterband except for  $\Delta\epsilon_{\text{tot}} = 0.5\%$ , a test which did not fail after 446,630 cycles. The points in the scatterband obey a Coffin-Manson-type relationship with

$$N_f = 1.58 \times 10^{-8} \Delta\epsilon_{\text{tot}}^{-5.65}, \quad (1)$$

where  $N_f$  is the number of cycles to failure and  $\Delta\epsilon_{\text{tot}}$  is the total strain range. An implication of the large exponent (for true Coffin-Manson behaviour it would be  $-2.0$ ) is that the life is propagation controlled. Since the stresses induced at 600°C for all strain ranges investigated are elastic, it is possible to express the behaviour in a Basquin relationship by converting the strain variable to stress. In this case,

$$N_f = 3.04 \times 10^{-13} \Delta\sigma^{-5.65}, \quad (2)$$

where  $\Delta\sigma$  is the stress amplitude,  $\Delta\sigma_{\text{tot}}/2$ , in MPa. The exponent in the Basquin relationship can be very roughly correlated with the  $m$  value in the Paris regime of fatigue crack growth, and the value obtained correlates reasonably well with values measured for nickel-base alloys.

Table 1: Testing matrix and fatigue results.

Total Strain Range (%)	Precracked?	Cycles to Failure	Notes
0.5	No	446,630+	No failure or crack initiation.
0.7	No	17,258	
0.7	No	16,977	
1.0	No	3,800	
1.0	No	1,853	
1.2	No	2,094	
1.4	No	477	
0.5	Yes (Nar.)	315,245+	No failure or crack growth.
0.6	Yes (Nar.)	330,384+	No failure or crack growth.
0.7	Yes (Nar.)	103,767+	No failure or crack growth.
0.7	Yes (Wide)	8,750	

A similar fracture morphology was observed for all failed specimens (Figure 2). All showed an initiation zone (no single point could be identified as an initiation site) on the outside surface of the specimen, a region of non-crystallographic crack growth perpendicular to the applied stress (mode I crack, stage II growth) in the vicinity of the initiation zone, and a progressive change to crystallographic crack growth on  $\{111\}$  planes (stage I growth) with increasing crack length away from the initiation zone. The transition from stage II to stage I growth was characterized by increasing surface roughness and an increased tendency for crack branching along  $\{111\}$  planes as the crack length increased. Figure 3 shows a typical initiation zone and stage II crack growth. The effect of increasing strain range for as-aged specimens on the fracture features was to cause a general increase in the surface feature

roughness in the region of stage II growth. There was no distinct trend in the relative amounts of stage II and stage I crack growth as a function of strain range.

The coating failure features were similar for all strain ranges. A typical micrograph is shown in Figure 4. Failure occurred by intergranular fracture in the fine-grained outer region of the coating, by transgranular cleavage in the coarser-grained central zone of the coating, and by separation along carbide-matrix interfaces in the coating transition zone. These features suggest that the coating has failed in a brittle manner rather than by a fatigue process. Secondary coating cracks and fatigue cracks were observed for all strain ranges (except 0.5%). The secondary cracks were similar to the primary crack in that fatigue initiation occurred at a coating crack and subsequent propagation was perpendicular to the applied stress. Extensive oxidation along the crack wake surfaces was not observed, nor was any significant plastic deformation ahead of the crack.

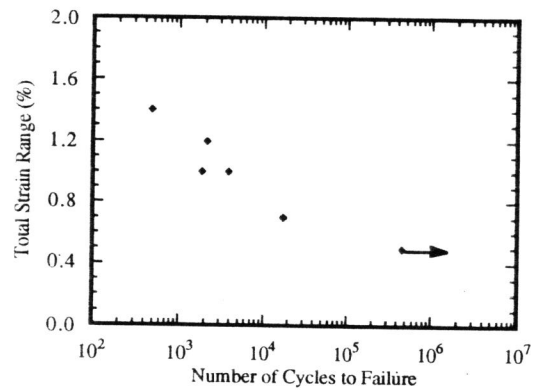


Figure 1:  $\epsilon$ -N curve for as-aged specimens.

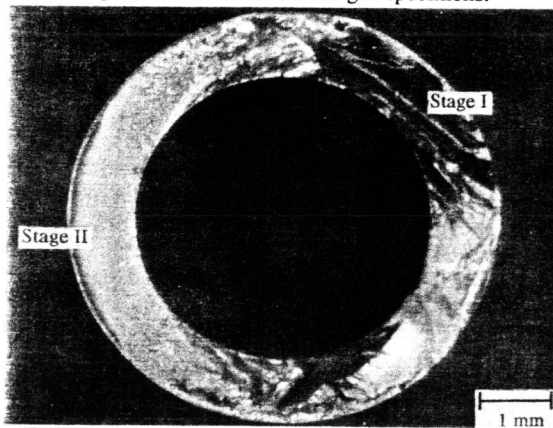


Figure 2: Low-magnification view of typical fracture morphology.

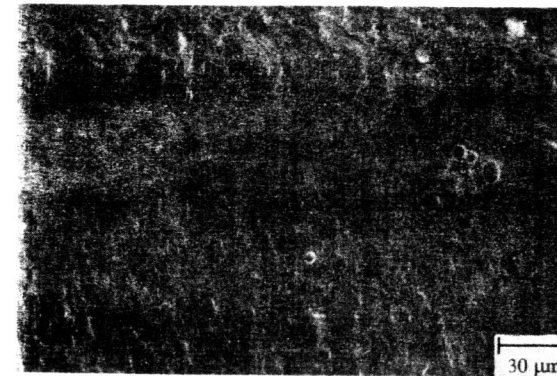


Figure 3: Typical initiation zone and stage II crack growth.

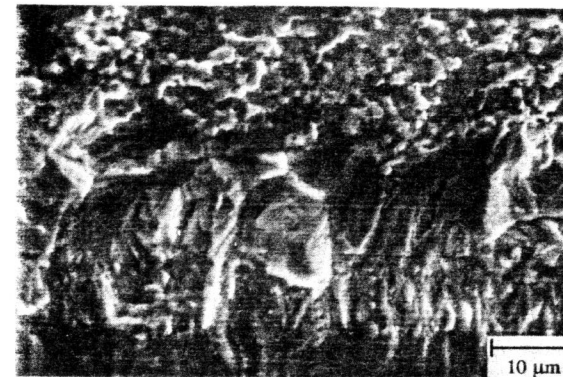


Figure 4: Coating failure features.



Figure 5: Oxide-filled narrow coating pre-crack.

**Pre-Cracked Specimens.** Room temperature precracking of the specimens produced apparently contradictory results. No crack growth after more than 100,000 cycles was observed from narrow precracks for specimens tested at total strain ranges of 0.5%, 0.6%, and 0.7%. Examination of metallographic cross-sections of these specimens revealed that the precracks were filled with oxide (Figure 5). However, failure of a specimen with wide precracks tested at a strain range of 0.7% did occur. The fracture morphology was similar to that observed in the as-aged tests. In particular the features of coating failure were the same, which further implies that coating failure of non-precracked (as-aged) specimens occurred in a brittle fashion.

## DISCUSSION

**Mechanisms of Crack Initiation and Growth.** The results obtained enable mechanisms of crack initiation and growth for this coating-substrate system at 600°C to be identified. Crack initiation for as-aged specimens tested at total strain ranges greater than or equal to 0.7% occurs via brittle cracking of the coating during the first fatigue cycle or cycles, as evidenced by the fracture surfaces and the morphology of secondary cracking. That brittle cracking of the coating would occur immediately is expected for total strain ranges at and greater than 1.0%, as the fracture strain of the coating (0.45%) is less than the maximum tensile strain applied in the fatigue cycle (0.5%). Brittle cracking of the coating would not be expected to occur for the specimen tested at a total strain range of 0.7%, since the maximum applied strain (0.35%) is less than the measured fracture strain. However, the similarity of the fracture surfaces, the existence of secondary coating cracks, and the fact that the life at 0.7% lies within the scatterband of the data for the higher strain ranges points to a similar fracture mechanism. Totemeier et al. (1993) previously observed that coating fracture in this coating – substrate system initiates at surface defects in the coating. These defects will have a statistical distribution of sizes; therefore it is possible that one or two defects of a critical size for cracking during fatigue at a total strain range of 0.7% will exist, leading to coating-initiated fatigue failure. Observation of fatigue crack growth from a brittle coating crack at a strain less than the observed fracture strain of the coating highlights a difficulty with utilizing such values, which generally refer to the presence of several visible cracks. Here it is apparent that fatigue is sensitive to the worst defect present.

The question of crack growth must also be considered. The principal problem with the observed growth mode is that crack growth in uncoated single crystal blade alloys is generally observed to occur in a crystallographic stage I manner at temperatures below around 750°C. There are two possible explanations for the observed behaviour in the coated system. First, it is possible that the fatigue crack growth behaviour for the substrate does show initial stage II growth at 600°C followed by a change to crystallographic growth at higher crack length/ $\Delta K$ . Such transitions from stage II to stage I growth have been observed by Leverant and Gell (1975), Henderson (1992), and Telesman and Ghosn (1988). The stage II behaviour in these cases has been associated with: (i) the frequency dependence of the homogeneous – heterogeneous deformation transition (Leverant and Gell), and (ii) dislocation activity and fatigue crack growth occurring primarily in the weaker  $\gamma$  matrix channels (Henderson, Telesman and Ghosn).

The second possibility is that the geometry of the initial defect (a coating crack) constrains crack growth such that stage II growth is observed. This is a possibility because the coating-

initiated crack is one which is short and extremely sharp even on a microscopic level (see Figure 5). Therefore the process of initiation of a fatigue crack from a machined notch (as with standard fatigue tests on uncoated alloys) is short-circuited. A fatigue crack can grow from the coating crack without the formation of relatively large planar slip bands. The initial plastic zone size in front of the crack tip is small (a fracture mechanics based estimate gives two microns for initial crack growth at a total strain range of 0.7%). As the crack grows and the stress intensity increases, the plastic zone size increases to a point where the crack is no longer constrained and crystallographic growth results.

It is not yet clear which of these possibilities is more likely. Further tests on the uncoated substrate (notched and un-notched) need to be performed to characterise its behaviour fully.

**Effects of Pre-Cracking.** The behaviour of the precracked tests can be explained in terms of oxide-induced crack closure (Suresh et al., 1981). Exposure to the ambient environment during heating of the specimens results in oxidation of the crack wake surfaces. Further build-up of the oxide occurs during testing due to fretting in the compressive half of the fatigue cycle. For narrow precracks, where the crack width is less than one micron, the formation and build-up of oxide will result in premature meeting of the crack wake surfaces and a reduction in the effective stress intensity range at the crack tip. For the lower strain ranges tested (0.7% and below) the initial stress intensity range of a coating precrack is close to the measured threshold value for the substrate alloy. The initial applied  $\Delta K$  at a strain range of 0.7% is  $5.1 \text{ MPa}\sqrt{\text{m}}$  while the measured threshold value is  $3.9 \text{ MPa}\sqrt{\text{m}}$ . Therefore a small reduction in effective stress intensity would prevent crack growth, as was observed for tests at 0.7% and below. However, premature contact of the crack surfaces does not occur for wide precracks (crack width approximately  $3 \mu\text{m}$ ), the applied stress intensity is not reduced, and crack growth results.

Two questions arise in consideration of the above results. The first is: why don't new cracks form at the strain range of 0.7%? If the precracks are effectively stopped, the specimen should behave as if it had not been precracked (*i.e.* failure in  $\sim 17,000$  cycles). The explanation lies in the process of precracking: new cracks do not form because cracks will already have formed at all defects large enough to result in coating fracture up to the highest strain level imposed during precracking. Areas between the precracks will therefore not contain any large defects, and formation of new cracks upon loading to the maximum strain of 0.35% (total strain range of 0.7%) in the fatigue cycle will not occur. The second question concerns cracks which have formed in the first fatigue cycles in non-precracked specimens at a total strain range of 0.7%. Should they not have become blocked with oxide? Here the critical factor is that the cracks are formed at temperature during the test and not at room temperature. Cracks formed during the test will grow immediately and not have time to form an oxide thick enough to prevent crack growth. Precracks formed at room temperature generally have several hours during specimen heat-up and stabilisation to oxidise.

## CONCLUSIONS

The fatigue behaviour of an aluminide coated single crystal nickel-base superalloy has been observed at 600°C. Brittle cracking of the coating was found to occur for total strain ranges at and greater than 0.7%. The coating cracks then served as initiation sites for subsequent fatigue crack growth in the substrate. The fatigue cracks initially grew in a stage II manner

perpendicular to the applied stress, and a transition to crystallographic stage I cracking along {111} planes occurred at longer crack lengths. It is not clear whether the initial stage II growth was associated with more homogeneous deformation behaviour of the substrate at lower stress intensity ranges or the geometric constraint of the coating-initiated fatigue cracks. The behaviour of specimens with coating pre-cracks formed at room temperature varied depending on the width of the pre-cracks. No crack growth was observed from specimens with narrow pre-cracks. This resulted from due crack closure caused by precrack oxidation during specimen heating and subsequent fretting of the crack surfaces during the compressive half of the fatigue cycle. However, no closure effects were observed for wide precracks - fatigue cracks grew from the precracks resulting in failure of the specimen.

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