Isothermal Fatigue of an Aluminide-Coated Single-Crystal Superalloy: Part II. Effects of Brittle Precracking

T.C. TOTEMEIER, W.F. GALE, and J.E. KING

The effect of brittle coating precracking on the fatigue behavior of a high-activity aluminide-coated single-crystal nickel-base superalloy has been studied using hollow cylindrical specimens at test temperatures of 600 °C, 800 °C, and 1000 °C. Three types of precrack were studied: narrow precracks formed at room temperature, wide precracks formed at room temperature, and narrow precracks formed at elevated temperature. The effect of precracking on fatigue life at 600 °C was found to depend strongly on the type of precrack. No failure was observed for specimens with narrow room-temperature precracks because of crack arrest *via* an oxidation-induced crack closure mechanism, while the behavior of wide precracks and precracks formed at elevated temperature mirrored the non-precracked behavior. Crack retardation also occurred for narrow room-temperature precracks tested at 800 °C—in this case, fatigue cracks leading to failure initiated in a layer of recrystallized grains on the inside surface of the specimen. A significant reduction in fatigue life at 800 °C relative to non-precracked specimens was observed for wide precracks and elevated temperature precracks. The presence of precracks bypassed the initiation and growth of coating fatigue cracks necessary for failure in non-precracked material. No effect of precracking was observed at 1000 °C.

I. INTRODUCTION

THIS article, the second of two parts, reports on an extension of a study on the isothermal fatigue of an aluminide-coated nickel-base superalloy.[1] In the present work, the effect of brittle monotonic precracking of the coating prior to fatigue testing was investigated. Precracking was examined both as a means of further exploring and verifying the mechanisms of fatigue in the non-precracked condition and as a simulation of possible cracking of the coating due to the application of high strains at low temperatures, as may occur in an out-of-phase thermomechanical fatigue (TMF) cycle.

Thermomechanical fatigue provides a closer simulation of the actual strain-temperature cycle that the coating and substrate experience in an engine environment than does isothermal fatigue. Unfortunately, testing of this type is more difficult to perform, and the results are harder to interpret. Coatings have shown the most detrimental effect on substrate TMF behavior when out-of-phase strain-temperature loops are used, *i.e.*, when the tensile strain is at a maximum when the temperature is at a minimum. In this case, brittle coating fracture at the lowest temperature leads to early fatigue crack initiation in the substrate and reduced life. As described by Strangman and Hopkins,[2] coating cracking can be assisted by compressive creep of the coating at high temperatures of the cycle. More recently, attempts have been made to relate isothermal and thermomechanical fatigue by using a "bithermal" cycle in which strains were applied isothermally at two different temperatures in each cycle.[3] Again, a detrimental coating effect was observed when a tensile strain was applied at the lower temperature.

In this study, isothermal fatigue tests at 600 °C, 800 °C, and 1000 °C were performed on high-activity aluminide-coated nickel-base superalloy single crystals. These temperatures were chosen to be below, close to, and well above the coating ductile-brittle transition temperature (DBTT). The coatings were precracked by monotonic loading prior to fatigue testing. Three different types of precrack were examined: narrow precracks formed at room temperature by loading to just beyond the coating fracture strain, wide precracks formed at room temperature by loading to a substantial plastic strain, and narrow precracks formed at an elevated temperature. The precracks formed at temperature were intended to simulate possible coating cracking in an out-of-phase TMF test and to provide some link between behavior observed in TMF and isothermal tests.

II. EXPERIMENTAL PROCEDURES

The single-crystal nickel-base superalloy substrate and high-activity aluminide coating used in this series of tests were identical to those previously described in detail in part I.[1] The dimensions of the ⟨100⟩-oriented low-cycle fatigue test specimens tested are also found in part I.

The aluminide coatings in this series of tests were monotonically precracked prior to fatigue testing. Three different types of precrack were generated. Narrow precracks were formed at room temperature by loading the specimen to an elastic strain of 0.7 pct, not significantly greater than the coating fracture strain of around 0.4 pct. Precracking of this type was intended to simulate possible cracking of the coating at room temperature. Wider precracks were formed at room temperature by loading the specimen to a strain of 2.0 pct. At this strain, plastic deformation of the substrate separates the crack surfaces after unloading. Specimens

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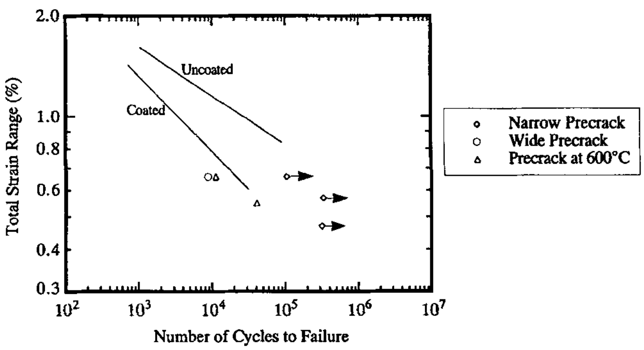

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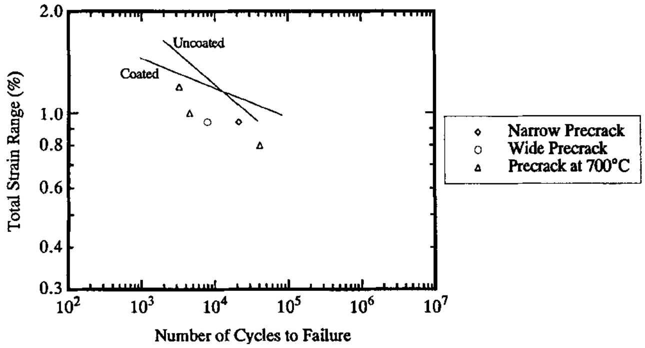


(*b*)

Fig. 1—Number of cycles to failure as a function of applied total strain range: (*a*) 600 °C and (*b*) 800 °C (baseline data from Ref. 1 shown as lines).

Table I. Modified Basquin Equation Parameters

Test Condition	σ_f' (MPa)	b
600 °C, coated	2940	−0.21
600 °C, uncoated	2810	−0.17
600 °C, precracked	1382	−0.14
800 °C, coated	1137	−0.07
800 °C, uncoated	2807	−0.16
800 °C, precracked	1758	−0.14

with wide precracks were tested for comparison with narrow precracked specimens. Some specimens were precracked at an elevated temperature, either 600 °C for fatigue tests at 600 °C or 700 °C for fatigue tests at 800 °C and 1000 °C. The temperature 700 °C lies just below the coating DBTT, enabling brittle cracking of the coating and also minimization of the time from precracking to fatigue testing in order to eliminate any oxidation-induced crack closure effects. All elevated temperature precracks were narrow, formed by loading to a strain of 0.7 pct.

Fully reversed ($\varepsilon_{min}/\varepsilon_{max} = -1$) isothermal fatigue tests on the precracked specimens were performed in total extension control at three temperatures: 600 °C, 800 °C, and 1000 °C. The temperatures were chosen to correspond to three regimes of expected behavior. At 600 °C, the coating is brittle, and the substrate is expected to show predominantly crystallographic stage I fatigue crack growth. At 800 °C, the coating is ductile, but the substrate is still expected to show crystallographic crack growth. At 1000 °C, the coating is ductile, and the substrate is expected to show noncrystallographic stage II crack growth.

Specimen temperature was maintained to ±1 °C using a cylindrical three-zone resistance furnace and was monitored using a Pt/Pt-Rh thermocouple. Testing employed a triangular waveform to give a constant strain rate of 3.5×10^{-3} s^{-1}, equivalent to 0.25 Hz at a total strain range of 0.7 pct. Fatigue life data were recorded as number of cycles to failure for all tests. The initial load response of all tests was noted. Total strain ranges investigated ranged from 1.2 to 0.5 pct. Post-test examinations of failed specimens were performed as described in part I.[1]

III. RESULTS

A. *Fatigue Lives*

Plots of applied total strain range *vs* number of cycles to failure graphically representing the fatigue life data at 600 °C and 800 °C are found in Figures 1(a) and (b), respectively. Baseline data obtained for uncoated and non-precracked specimens, presented in part I,[1] are also shown for comparison. Baseline data are shown as lines in Figures 1(a) and (b), while data for precracked specimens are shown as individual points. The data for the non-precracked specimens and specimens precracked at elevated temperature can be represented numerically using a modification of the Basquin relationship:[4]

$$\frac{\Delta\varepsilon_{tot}}{2} = \frac{\sigma_f'}{E}(2N_f)^b \qquad [1]$$

where $\Delta\varepsilon_{tot}$ is the applied total strain range, σ_f' is the fatigue strength coefficient, E is the elastic modulus, and b is the fatigue strength exponent. Ordinarily, a plasticity term also appears on the right-hand side of the equation; however, it is equal to zero for the elastic strains used in this study. Values of the Basquin parameters for each condition are given in Table I.

The effect of precracking on fatigue life at 600 °C was dependent on the type of precrack. Specimens with narrow room-temperature precracks did not fail at any of the three strain ranges investigated at 600 °C (0.47, 0.57, and 0.66 pct); non-precracked specimens failed at strain ranges at and greater than 0.6 pct. Specimens with wide room-temperature precracks or precracks formed at 600 °C had slightly lower fatigue lives than the non-precracked material, but the decrease was not statistically significant. The low σ_f' value obtained for specimens precracked at 600 °C appears to be an artifact of the small number of data points.

All precracked specimens tested at 800 °C had shorter fatigue lives than non-precracked specimens. The narrow room-temperature precracked specimen had a longer life at a strain range of 0.66 pct than the specimen with wide precracks; however, both had shorter lives than the non-precracked material. The life of the specimen with wide precracks was similar to that of specimens precracked at 700 °C. The curve fit to the data of specimens precracked at 700 °C had a much lower σ_f' than non-precracked uncoated specimens but it had a similar b value.

Only one specimen was available for fatigue testing at 1000 °C. The lives of this test (precracked at 700 °C) and the non-precracked specimen tested at the same strain range (1.0 pct) were similar. The non-precracked specimen failed after 2721 cycles, while the precracked specimen failed after 2452 cycles.

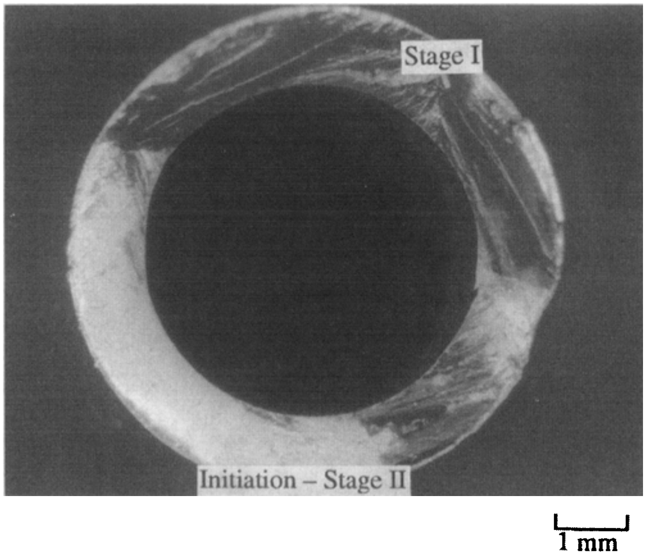


Fig. 2—Typical fracture morphology observed at 600 °C ($\Delta\varepsilon_{tot}$ = 0.66 pct, narrow 600 °C precrack).

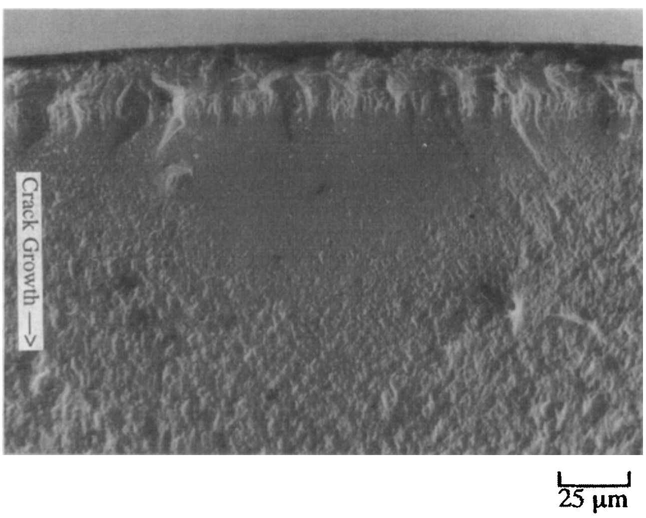


Fig. 3—Typical initiation zone and stage II growth features at 600 °C ($\Delta\varepsilon_{tot}$ = 0.55 pct, narrow 600 °C precrack).

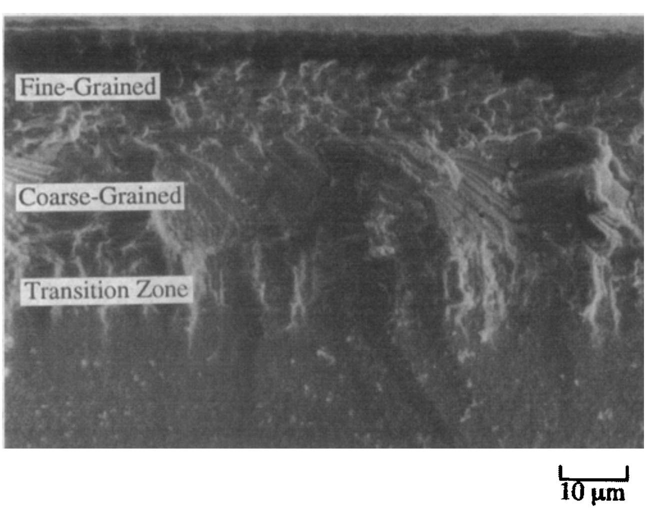


Fig. 4—Close-up of coating failure features at 600 °C ($\Delta\varepsilon_{tot}$ = 0.55 pct, narrow 600 °C precrack).

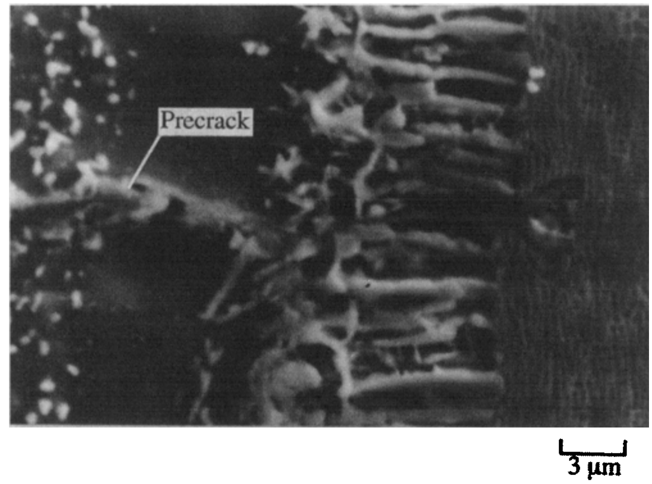


Fig. 5—Narrow room temperature precrack filled with oxide following testing at 600 °C ($\Delta\varepsilon_{tot}$ = 0.47 pct). Substrate is on the right-hand side of the micrograph.

B. *Failure Morphology*

1. *600 °C*

The failure morphology of precracked specimens fatigue tested at 600 °C was identical to that observed for non-precracked material. Initiation occurred in a broad zone along the coated surface. Crack propagation in the substrate was initially in a noncrystallographic stage II manner perpendicular to the applied stress with a subsequent transition to crystallographic crack growth along {111} planes at longer crack lengths (Figure 2). A typical initiation region and stage II growth in the substrate are shown in Figure 3. The fracture features of the coating were brittle in nature. Failure occurred by intergranular separation in the outer region of the coating, by transgranular cleavage in the coarser-grained central region, and by separation along carbide-matrix interfaces in the coating transition zone (Figure 4). Regions of distortion due to contact of the opposing surfaces during testing are also visible.

Examination of metallographic cross sections of both failed and run-out tests revealed extensive cracking of the coating along the entire gage length of the specimen. The cracks were distributed uniformly with a linear density of 5 to 8 mm; the crack density was the same for all types of precrack. The fracture features of cracks as observed in cross section were the same as observed on the fracture surface. Coating crack widths varied with precrack type, as expected. Narrow room-temperature precracks were filled with oxide following testing at 600 °C; the oxide width was 2 μm (Figure 5). The wide room-temperature precracks were 4- to 6-μm wide, and the precracks formed at 600 °C were 0.5-μm wide. Substantial oxide buildup was not observed for these types of precrack.

Secondary fatigue cracks which had grown from the coating cracks into the substrate were observed in the cross sections of failed specimens, *i.e.*, specimens with wide room-temperature precracks or narrow elevated temperature precracks. The cracks were very fine and oriented perpendicular to the applied stress. No planar slip bands were observed either in the crack wake or ahead of the crack tip. No secondary fatigue crack growth was observed from narrow room-temperature precracks at all strain ranges.

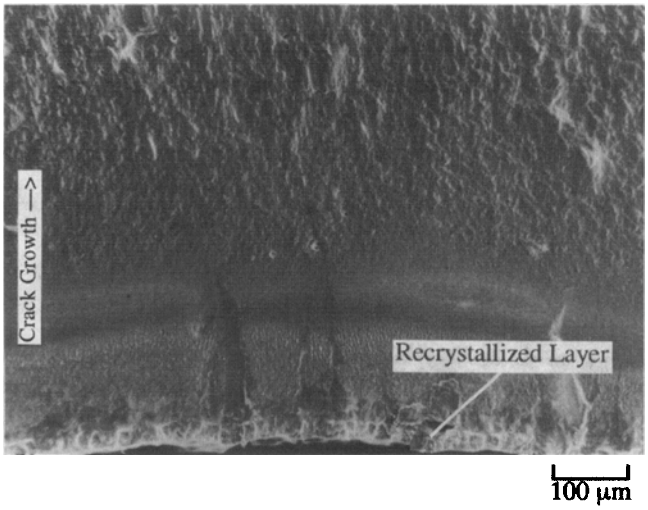


Fig. 6—Fatigue crack initiation at a recrystallized layer on the inside specimen surface at 800 °C ($\Delta\varepsilon_{tot}$ = 0.94 pct, narrow room-temperature precrack).

2. *800 °C*

The failure morphology of precracked specimens tested at 800 °C was again similar to the non-precracked morphology. As at 600 °C, initiation occurred at the coated surface with initial stage II crack propagation in the substrate and a transition to crystallographic crack growth at longer crack lengths. The specimen with narrow room-temperature precracks was one exception to this rule. For this test, the crack which led to failure initiated at the uncoated inside surface of the specimen at a thin layer of recrystallized grains (Figure 6), with some growth from the coated outside surface. The features of crack growth at 800 °C, both coating and recrystallization initiated, were similar to those at 600 °C but were somewhat rougher and more oxidized.

Coating cracks in precracked specimens fatigue tested at 800 °C showed two morphologies. The first type was widely spaced cracks which took a relatively straight path through the coating (Figure 7(a)). The second type was groups of narrowly spaced cracks which were found at locations far from the widely spaced cracks. These cracks had a contorted morphology (Figure 7(b)) similar to secondary coating cracks observed in non-precracked specimens. All coating cracks were oxidized. As also shown in Figures 7(a) and 7(b), stage II crack growth into the substrate for all coating cracks was accompanied by oxidation of the crack wake.

A layer of recrystallized grains was observed in the substrate adjacent to the inside surface of some specimens. Secondary fatigue cracks initiated at this recrystallized layer during testing at 800 °C for all specimens in which the recrystallized zone was present. Crack growth from the recrystallized zone led to failure of the specimen with narrow room-temperature precracks. In other tests, the amount of crack growth was small and did not significantly affect the overall fatigue life.

3. *1000 °C*

Two large (greater than 2 mm in length) primary fatigue cracks were observed for the precracked specimen fatigue tested at 1000 °C. Both cracks had an oxidized stage II morphology identical to that of non-precracked specimens and consistent with the high testing temperature and low substrate yield strength. The secondary coating crack morphology was also identical to that of non-precracked specimens. Nearly all were arrested by severe oxidation at the coating-substrate interface, as shown in Figure 8.

IV. DISCUSSION

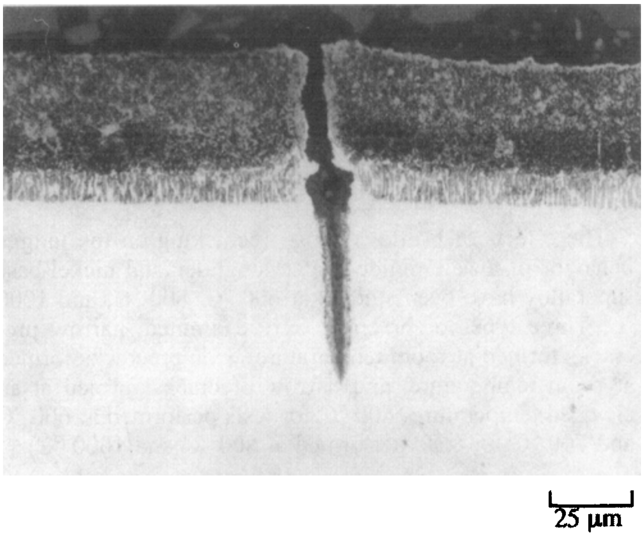
The fracture morphologies and mechanisms of fatigue crack growth for this coating-substrate system have been discussed previously in part I.[1] Therefore, only the specific effects of precracking on the observed failure mechanisms and fatigue lives will be discussed in this article.

A. *Effects of Precracking—600 °C*

The apparently contradictory behavior of the fatigue tests performed at 600 °C can be explained in terms of oxidation-induced crack closure.[5] Exposure to the ambient air environment during heating of the specimens resulted in oxidation of the crack wake surfaces for precracks formed at room temperature, both narrow and wide. Further buildup of the oxide occurred during testing due to fretting in the compressive half of the fatigue cycle. For narrow room-temperature precracks, the formation and buildup of oxide resulted in premature meeting of the crack wake surfaces and a reduction in the effective stress intensity range at the crack tip. The buildup of oxide is clearly apparent in Figure 5. The crack width increased during testing from 0.5 μm (assuming that the initial width of room-temperature precracks was equal to that of precracks formed at 600 °C) to 2 μm. For the strain ranges tested (0.7 pct and below), the initial stress intensity range of a coating precrack was close to the measured threshold value for the substrate alloy. The initial ΔK ahead of a 45- to 50-μm coating precrack at a total strain range of 0.7 pct was 5 MPa$\sqrt{m}$ (calculated using the relation $\Delta K = Y\Delta\sigma\sqrt{\pi a}$, with Y = 1.11 for a circumferential coating crack,[6] $\Delta\sigma = \Delta\varepsilon_{tot}E/2$, and a = 50 μm), while the measured threshold value was 4 MPa$\sqrt{m}$[7] (the threshold value was determined at a high R ratio to ensure a fair comparison with the threshold likely to be observed for a short coating precrack). Therefore, a small reduction in effective stress intensity would prevent crack growth, as was observed.

A question arises in the consideration of these results. Why were no new coating cracks formed at the strain range of 0.7 pct? If the precracks were effectively stopped, the specimen should have behaved as if it had not been precracked, *i.e.*, the coating should have fractured again upon reaching the maximum tensile strain in the first fatigue cycle.[1] The explanation lies in the process of precracking: new cracks did not form because cracks will form during precracking at all defects large enough to result in coating fracture up to the highest strain level imposed. Areas between the precracks, therefore, will not contain any large defects, and formation of new cracks upon loading to the maximum strain of 0.35 pct (total strain range of 0.7 pct) in the fatigue cycle will not occur.

A reduction in stress intensity did not occur for either wide precracks or precracks formed at 600 °C. In the case of wide precracks, the crack width (4 to 6 μm) was suffi-



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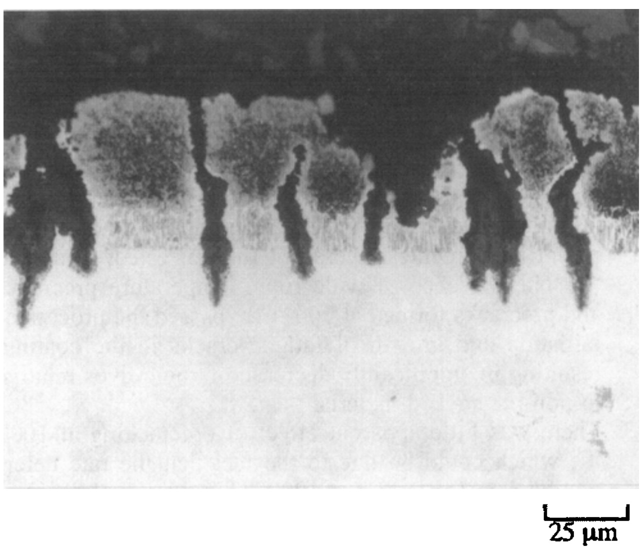

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Fig. 7—Secondary coating cracks observed at 800 °C ($\Delta\varepsilon_{tot}$ = 0.94 pct, narrow room-temperature precrack): (*a*) widely spaced precracks and (*b*) narrowly spaced fatigue cracks. Substrate is toward the bottom of the micrograph.

cient to keep the crack wake surfaces well separated during the tensile half of the fatigue cycle, eliminating any possibility of effective stress intensity reduction due to premature contact. The oxidation which occurred during specimen heatup and stabilization was the key difference between cracks formed at room temperature and cracks formed at 600 °C. Crack growth commenced immediately following the formation of elevated temperature precracks; there was not enough time for oxidation sufficient to retard the crack to occur.

The similarity of fracture morphology and the equality of fatigue lives for precracked specimens which failed and non-precracked specimens lend further credence to the suggestion that fatigue crack initiation occurs in non-precracked specimens during fatigue testing at 600 °C by brittle coating cracking in the first fatigue cycle, as described in part I.[1] In particular, the coating failure features observed on the fracture surfaces were identical. The small,

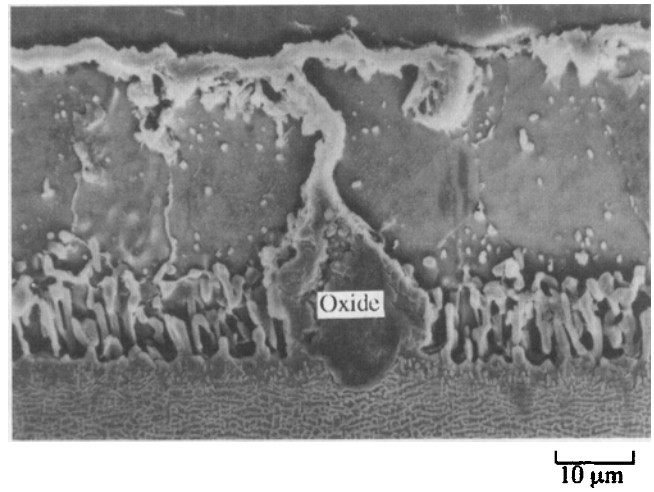

Fig. 8—Typical secondary crack at 1000 °C ($\Delta\varepsilon_{tot}$ = 0.66 pct, narrow 1000 °C precrack).

perhaps insignificant, reduction in fatigue life which was observed on precracking could be related to the larger, more effective initial defect generated by the precracking process of loading to a tensile strain of 0.7 pct. In non-precracked specimens tested at equivalent strain ranges, the maximum tensile strain was only 0.3 to 0.35 pct. At the lower strain ranges, fewer and smaller (in circumferential length) cracks will be generated.

B. *Effects of Precracking—800 °C and 1000 °C*

A lowering of the effective stress intensity due to oxide-induced crack closure was also expected for narrow room-temperature precracks fatigue tested at 800 °C. However, the cracks appeared only to be retarded and not fully arrested (as at 600 °C), as shown by the secondary crack growth into the substrate from the precracks. The difference between the initial stress intensity range and the threshold value was higher than at 600 °C for the narrow room-temperature precrack test, which was performed at a total strain range of 0.94 pct. The initial stress intensity range ahead of a coating precrack was approximately 6 MPa$\sqrt{m}$ (calculated in the same fashion as earlier), while the threshold value has been measured as 3 MPa$\sqrt{m}$.[7] Therefore, a greater closure effect would be needed to arrest crack growth from coating precracks fully.

However, the reduction in precrack driving force due to oxide-induced crack closure for narrow room-temperature precracks was sufficient to result in the dominance of cracks which initiated at a recrystallized layer on the inside surface of the specimen. This layer was formed as a consequence of both machining damage to the substrate during honing of the central hole and the 1100 °C diffusion treatment following aluminizing. The machining provided the strain energy needed for recrystallization, which subsequently occurred during the high-temperature exposure of the diffusion treatment.[8] The grain boundaries of the recrystallized layer are very susceptible to crack growth at 800 °C, and fatigue crack initiation will occur quickly.[9]

As at 600 °C, the wide precracks and precracks formed at 700 °C were not subject to crack retardation during fatigue testing at 800 °C, for the same reasons mentioned previously for 600 °C. For these tests, the crack leading to failure initiated at a coating precrack, and cracks which initiated at the recrystallized layer did not significantly influence the fatigue life. For both types of precrack, fatigue life was significantly lowered relative to the non-precracked material. For non-precracked material, initiation and crack growth in the coating were shown in part I[1] to occur by a fatigue process at 800 °C due to the large increase in coating ductility and the reduction in coating strength above the DBTT. In contrast, crack growth in the substrate can begin immediately at the start of the fatigue test when precracks are present.

It was clear from the crack morphologies that the two types of coating cracks observed following fatigue testing at 800 °C were precracks and "naturally" initiating coating cracks which grew by a fatigue process. The wide spacing, equivalent to the spacing observed for precracks in 600 °C tests (5 to 8 mm), of cracks which passed relatively straight through the coating marked them as precracks. Likewise, the cracks which formed during the test had a spacing and morphology similar to those observed in non-precracked specimens.[1] The location of coating fatigue cracks far from the precracks was due to the reduction in local stress in the coating adjacent to the precracks.

There was apparently little effect of precracking on the fatigue behavior at 1000 °C. Only one specimen was available for testing at this temperature. The fatigue life obtained could have been influenced by the presence of two major cracks, which could have reduced the stress intensity operative at each crack tip in the strain-controlled test. The morphology of secondary cracking was identical to that observed in tests of non-precracked specimens, with very little secondary crack growth in the substrate. This suggested that the rate determining step for failure at this temperature was the initial penetration of coating-initiated fatigue cracks into the substrate, whether from a coating crack or the uncoated surface. Apparently, both coating failure and crack growth in the substrate occur relatively quickly. In this case, little difference would be expected between the life of a specimen with precracks and one without, as observed.

C. *Applicability to TMF Testing*

The behavior of isothermal precracked tests can provide a useful link to TMF tests *via* the fracture morphologies observed. Comparison of a given TMF failure surface with those produced in isothermal precracked and non-precracked tests should enable us to identify at which temperature and in what manner damage accrued in the TMF test. For example, the presence of widely spaced secondary cracks in a cross section of a TMF test would indicate that the coating had been fractured at a low temperature, while narrowly spaced cracks would indicate the dominance of a high-temperature damage mechanism.

The reduction in fatigue life for tests in which the coating was cracked at temperature verifies that brittle coating cracking at low temperature in a TMF test is likely to lead to significant life reductions compared to cases in which cracking does not occur, *e.g.*, an in-phase cycle. The reduction also suggests that the coating can be more damaging in TMF tests than isothermal tests at high temperatures would indicate, highlighting a problem with using isothermal tests at a maximum expected temperature to predict life under a TMF cycle. Further tests at 1000 °C are needed to explore the effects at this temperature, a typical peak temperature used in TMF testing.

V. CONCLUSIONS

The effects of brittle coating precracking on the fatigue behavior of an aluminide-coated single-crystal nickel-base superalloy have been studied at 600 °C, 800 °C, and 1000 °C. Three types of precrack were examined: narrow precracks formed at room temperature, wide precracks formed at room temperature, and narrow precracks formed at an elevated temperature (600 °C for tests performed at 600 °C and 700 °C for tests performed at 800 °C and 1000 °C).

1. The effect of precracking on fatigue tests carried out at 600 °C was dependent on the type of precrack. No crack growth or failure occurred from narrow precracks formed at room temperature for any of the strain ranges investigated, due to oxidation-induced crack closure. No significant closure effect was present for wide room-temperature precracks or precracks formed at 600 °C. In both cases, fatigue lives were slightly lower than non-precracked behavior.
2. Retardation of crack growth from narrow room-temperature precracks also occurred during fatigue testing at 800 °C; however, the lower fatigue threshold at 800 °C meant that precracks were not fully arrested, as at 600 °C. The presence of wide room-temperature precracks and precracks formed at 700 °C bypassed the process of initiation and growth of fatigue cracks in the coating, resulting in significantly decreased fatigue lives relative to non-precracked material.
3. There was little apparent effect of precracking at 1000 °C, which could be due to the fact that the rate determining step for failure at 1000 °C is the initial penetration of a coating-initiated crack into the substrate.

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