The Low Cycle Fatigue Behavior of a Plasma-Sprayed Coating Material

Lewis Research Center
Cleveland, Ohio

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THE LOW CYCLE FATIGUE BEHAVIOR OF A PLASMA-SPRAYED COATING MATERIAL

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

SUMMARY

Single crystal nickel-base superalloys employed in turbine blade applications are often used with a plasma sprayed coating for oxidation and hot corrosion resistance. These coatings may also affect fatigue life of the superalloy substrate. As part of a larger program to understand the fatigue behavior of coated single crystals, fully reversed, total strain controlled fatigue tests were run on a "free standing" NiCoCrAlY coating alloy, PWA 276, at 0.1 Hz. Fatigue tests were conducted at 650 °C, where the NiCoCrAlY alloy has modest ductility, and at 1050 °C, where it is extremely ductile, showing tensile elongation in excess of 100 percent. At the lower test temperature, deformation induced disordering softened the NiCoCrAlY alloy, while at the higher test temperature cyclic hardening was observed which was linked to gradual coarsening of the two phase microstructure. Fatigue life of the NiCoCrAlY alloy was significantly longer at the higher temperature. Further, the life of the NiCoCrAlY alloy exceeds that of coated, [001]-oriented PWA 1480 single crystals at 1050 °C, but at 650 °C the life of the coated crystal is greater than that of the NiCoCrAlY alloy on a total strain basis.

INTRODUCTION

Overlay MCrAl coatings have been found to provide excellent surface protection for gas turbine blades and vanes. An important advantage of overlay coatings is the inherent compositional flexibility which permits tailoring of the coating chemistry to give optimal oxidation resistance and coating substrate compatibility (ref. 1). This compositional flexibility allows the addition of active elements, such as Y which improves the adherence of the oxide scale during thermal cycling (ref. 2) for improved oxidation resistance.

While the beneficial effects of the MCrAlY overlay coatings on the oxidation behavior of superalloy substrates are well understood, the effects of these coatings on the fatigue behavior of the superalloy substrate is less certain. As part of an overall program to address fatigue problems, especially thermochemical fatigue, of coated single crystal superalloy components, a fundamental study of the fatigue behavior of "free standing" NiCoCrAlY was undertaken. Due to the tremendous variability in tensile strength and ductility between room temperature and maximum application temperature (ref. 3), fatigue tests were performed at 650 °C where NiCoCrAlY exhibits high strength and moderate ductility, and 1050 °C where NiCoCrAlY shows extremely high ductility and low strength. It was also thought that the fatigue-creep-environment interactions would differ significantly at the two test temperatures. The isothermal fatigue behavior of the NiCoCrAlY will be compared to that of PWA 1380 [001] single crystals coated with NiCoCrAlY. These fatigue life comparisons will be made on a total strain basis, as the strain levels in the coating of actual hardware follow that of the substrate for the most part.
MATERIAL AND PROCEDURES

The NiCoCrAlY coating alloy studied here, PWA 276, was developed by Pratt and Whitney Aircraft. The composition of the alloy in weight percentage was 13.6 Al, 17.3 Cr, 20.3 Co, 0.5 Y, 0.01 C, and balance Ni. "Free standing" NiCoCrAlY was produced by a low pressure plasma spray process by a commercial vendor in the form of plates about 8 x 15 x 0.09 cm thick. The plates were then heat treated in air at 1100 °C for 4 hr followed by 870 °C for 32 hr. This heat treatment simulates that given PWA 276 coated single crystal hardware used in Pratt and Whitney gas turbine engines.

Miniature low cycle fatigue specimens shown in figure 1 were machined from the plates by stress-free grinding to minimize surface damage. The gauge section and radius were given an 8 RMS finish by lapping parallel to the stress axis.

Total axial strain controlled fatigue tests were run in air at 650 and 1050 °C on a closed-loop, servohydraulic test machine. A sinusoidal, 0.1 Hz waveform with a maximum strain/minimum strain ratio of -1 was employed. At 650 °C a three-zone clam shell furnace was used for specimen heating, and axial strain measurements were obtained using a self-supporting, 12.5 mm gauge length extensometer. At 1050 °C, in deference to problems with specimen/grip seizure, a kW radio frequency induction generator was used to heat the specimens. Due to the material's very low strength at this temperature, it was not possible to use the extensometer for direct strain measurement across the gauge length, as the quartz probes marred the specimen surface. Instead the grip displacement was used to calculate specimen strain. A traveling telescope was used to calibrate grip displacement and specimen strain at temperature, by measuring the relative movement of small surface features, i.e. dirt or tiny scratches. Load-time and strain-time traces were continuously recorded with a two pen strip chart recorder, while hysteresis loops were periodically recorded with an X-Y recorder.

Failed specimens were examined with a scanning electron microscope to determine the modes of crack initiation and propagation. In addition, metallographic sections of selected specimens, from both failed and interrupted tests, were prepared to augment the SEM findings and disclose any microstructural changes.

RESULTS

Microstructure

The microstructure of the as-received NiCoCrAlY plates exhibits about 3 percent porosity, with an average pore size of approximately 20 μm. This represents about twice the level of porosity observed in thin coatings deposited by the same vendor on superalloy components for gas turbine engines. Although current production of commercial plasma coatings is well developed, some microporosity is found even in thin coatings, and the process was certainly not optimized for the production of thick plates.

Etching of the as-received material revealed spherical powder particles of the NiCoCrAlY powder which were not melted in the plasma spraying operation. After heat treatment the microstructure of the plates was very uniform, figure 2,
with a fine grained two-phase microstructures consisting of the dark $\beta$ phase (N1A1) and the $\gamma$ phase (Ni-rich solid solution). The grain size of the NiCoCrAlY alloy was less than 3 $\mu$m, which is comparable to that found in thin coatings applied to superalloy components.

Tensile Behavior

The monotonic tensile properties of the NiCoCrAlY at 650 and 1050 °C are summarized in table I. At 650 °C the NiCoCrAlY exhibited high strength and moderate ductility, but at 1050 °C the material is very weak and exhibits superplastic behavior at the tensile strain rate employed. A more extensive compilation of data on the tensile behavior of the NiCoCrAlY can be found elsewhere (ref. 3).

Fatigue Behavior

The vast difference in strength and ductility at the two test temperatures is also reflected in the stress-strain response observed during fatigue testing. Figure 3 shows the 650 and 1050 °C hysteresis loops of the NiCoCrAlY at half life for $\Delta e = 0.01$. Note that only a small fraction of the total strain range is elastic at 1050 °C. This was case for all tests run at 1050 °C.

At 650 °C extensive softening of the NiCoCrAlY was observed in all fatigue tests. The actual softening response is shown in figure 4 for three strain ranges. In each instance the softening started from the onset of the test and continued at a measurable rate until failure. The NiCoCrAlY also exhibited a persistent compressive mean stress, varying from a high of 8 percent to a low of 1 percent of the total stress range for the highest and lowest strain ranges, respectively. The cyclic stress-strain response of the NiCoCrAlY at half life is presented in figure 5, and gives a cyclic strain hardening exponent of $m = 0.19$.

Fatigue life of the NiCoCrAlY and the NiCoCrAlY-coated PWA 1480 (ref. 4) at 650 °C is plotted in figure 6. Over the test range, the life of the NiCoCrAlY is less than that of the coated PWA 1480, on a total strain basis, and this difference increases at lower strain ranges. The elastic and inelastic life lines of the NiCoCrAlY are presented in figure 7. Also shown are the regression equations for the two life lines.

Unlike the softening observed at 650 °C, the NiCoCrAlY hardens profusely at 1050 °C, as seen in figure 8. Hardening was observed at all strain ranges and reaches a maximum near the end of the test. Failure is not abrupt at this temperature, as illustrated by the terminal "softening" in figure 8, which is related to stable crack growth produced by strain control. Within the accuracy of the stress measurements at 1050 °C, no consistent mean stresses were observed.

The 1050 °C fatigue life response of the NiCoCrAlY and NiCoCrAlY-coated PWA 1480 (ref. 4) are presented in figure 9 on a total strain basis. Over the range of tests performed, the NiCoCrAlY had a fatigue life about five times greater than that of the coated single crystal. The cyclic stress-strain response of the NiCoCrAlY is plotted in figure 10 and illustrates the immense
decrease in strength at this temperature, compare the coefficients of the regression equations, 166 MPa at 1050 °C versus 2705 MPa at 650 °C. Further, the cyclic hardening exponent, \( m = 0.22 \), is slightly higher than that observed at 650 °C, \( m = 0.19 \).

A comparison of the fatigue lives of the NiCoCrAlY, figure 11, shows the 1050 °C life to be significantly greater than that at 650 °C on a total strain basis. This difference becomes even greater when the comparison is made on an inelastic strain basis, as might be expected from ductility considerations.

**Microstructural Stability**

Accompanying the large variation in life are significant differences in microstructural stability at the two test temperatures. Significant grain growth and redistribution of the \( \gamma \) and \( \beta \) phases were observed at 1050 °C as seen in figure 12, however, at 650 °C the microstructure was stable even at the higher strain ranges. Porosity level was affected in a similar manner. A net reduction of porosity was observed in fatigue tests at 1050 °C, although large pores were present throughout the test. At 650 °C porosity content and distribution were virtually unaffected. Returning to figure 12, it is apparent that the applied strain has accelerated the grain growth process. Simple heating at 1050 °C has altered grain size and phase distribution, but not to the extent seen in interrupted fatigue tests of equivalent time. Further, porosity content and "remnant powder" structure are virtually unaffected by simple thermal treatment for times up to 24 hr.

Accompanying the microstructural instabilities at 1050 °C, surface "rumpling" and cracking were observed in the NiCoCrAlY, as seen in figure 13, while little if any surface damage is seen at 650 °C. Surface cracking at 1050 °C was readily observable at or shortly after half life.

**Fracture Modes**

Fracture initiation and propagation at 650 °C is characterized by the photomicrographs in figure 14. A singular, mixed mode initiation event was identified in most specimens, generally associated with a cluster of pores at or near the surface. The mode of crack propagation quickly became intergranular as was the final fracture event.

At 1050 °C the fracture process was quite different, multiple surface initiation sites were identified on all specimens, as seen in figure 15. Surprisingly, the mode of initiation and early propagation was transgranular. As shown in figure 16, the transgranular fracture mode extended to depths of 50 \( \mu \)m or more after which failure became intergranular. Close examination also discloses depletion of the \( \beta \) phase due to oxidation of aluminum, along the specimen surface and the crack faces. Also not the lack of large pores or cracks ahead of the main crack tip in figure 16.
DISCUSSION

650 °C Fatigue Behavior

As previously stated, cyclic softening of the NiCoCrAlY was observed at 650 °C. Possible causes of this softening include relaxation of an initial dislocation substructure, accelerated creep processes associated with enhanced vacancy generation, and disordering of the β phase and/or fine γ' in the γ phase. The existence of an initial dislocation substructure in cold worked material often leads to softening (ref. 5). Although the NiCoCrAlY was not cold worked, it was thought that the plasma spray process may have produced a significant dislocation substructure. However, subsequent TEM examination of the NiCoCrAlY showed very little dislocation substructure before testing. Also, any explanation based on accelerated creep rates appears to be incorrect as testing at 1.0 Hz, ten times faster than the base frequency, showed little if any changes in the rate or amount of softening. Disordering has been shown to produce cyclic softening in a number of materials including superalloys such as Waspaloy (ref. 6) and Inconel 718 (ref. 7). In these materials the disordering, caused by continual movement of dislocations in slip bands results in atomic rearrangement over very small distances. Local order and alloy strength can be restored by short time anneals since little long range movement of atomic species is required. Annealing the NiCoCrAlY at 760 °C for 10 min was found to restore the strength after softening had occurred, figure 17. Continued cycling of the NiCoCrAlY at 650 °C subsequently resoftened the material. Based on these observations it would appear that the softening of the NiCoCrAlY at 650 °C is caused by disordering of the β phase and/or fine γ' in the γ phase.

At 650 °C the strain-fatigue life relation for the NiCoCrAlY alloy was similar to that of other metallic materials in that the slopes of the inelastic and elastic life lines were about -0.6 and -0.1, respectively. The aspect of the fatigue life which was most surprising at 650 °C was the lack of scatter, as shown in any of the life plots. An explanation for this lack of scatter is probably related to the high level of porosity present in the NiCoCrAlY, which leads to a relatively large number of surface or near-surface initiation sites in each specimen. This could decrease the scatter associated with crack initiation.

At 650 °C the life of the NiCoCrAlY was less than that of the PWA 1480 coated single crystals. While this does not conclusively show fatigue cracks initiated in the coating on superalloy substrates are benign, it does indicate such flaws do not produce a rapid catastrophic failure of the superalloy substrate. This conclusion is also supported by limited comparison of coated and uncoated PWA 1480 fatigue tests which have essentially similar life at 650 °C, over the strain ranges studied herein.

1050 °C Fatigue Behavior

The cyclic hardening behavior of the NiCoCrAlY at 1050 °C was somewhat surprising in light of the cyclic softening observed at 650 °C. While softening associated with disordering is probably minimal at 1050 °C as the deformation is more homogeneous and the higher diffusion rates produce rapid reordering, the actual hardening process is probably produced by the rapid grain growth observed in these tests, as high temperature strength of
polycrystalline materials generally increase with increasing grain size. A further correlation between these two factors is illustrated by a detailed comparison of figures 8 and 12. In the lower strain range test little hardening is observed in the first hour of testing (N = 360 cycles) and very little grain growth is evident. From this point on the hardening rate and the rate of grain growth accelerate reaching a maximum near the end of the test. As previously stated the terminal "softening" is a result of significant cracking.

The increased fatigue life of the NiCoCrAlY at the higher test temperature is probably due in large part to sharply lower crack growth rates. This is especially important at large strains employed here where crack initiation is less difficult. Direct observations of cracks on the specimen surface at 1050 °C at half life support this concept. The diminished crack growth rates at elevated temperature are probably due primarily to the mode of crack growth, which is characterized by multiple transgranular cracking at 1050 °C, as opposed to the singular, intergranular crack mode observed at 650 °C. In addition crack tip blunting at 1050 °C, associated with creep deformation and oxide buildup, is probably retarding crack growth rates even further.

Comparison of the NiCoCrAlY and coated PWA 1480 has shown the fatigue life of the coating is far greater at 1050 °C. This is consistent with the observed failure mode in fatigue tests of the coated PWA 1480 (ref. 4) at 1050 °C, where internal crack initiation was identified as the source of failure in a majority of specimens. This finding is not at odds with the rapid rate of crack initiation in the NiCoCrAlY, as the time to initiate and propagate a 100 μm crack, the approximate coating thickness on the PWA 1480 specimens, is probably still greater than the observed life of the PWA 1480. Recall the life difference between the NiCoCrAlY and the coated PWA 1480 is greater than a factor of five.

CONCLUSIONS

Low cycle fatigue tests were run on a "free standing" NiCoCrAlY coating alloy at 650 and 1050 °C at strain ranges from 0.005 to 0.015.

At 650 °C:

(1) The NiCoCrAlY showed significant softening.

(2) This softening process was related to deformation induced disordering.

(3) Failure was characterized by singular surface or near-surface initiation sites of mixed mode, generally associated with porosity, followed by intergranular crack growth.

(4) Fatigue life of the NiCoCrAlY was significantly less than that of coated PWA 1480 [001] single crystals.

At 1050 °C:

(1) Strains were largely inelastic as the NiCoCrAlY was very weak but extremely ductile.
(2) The NiCoCrAlY displayed cyclic hardening and this process was linked to significant grain growth.

(3) Crack initiation and early propagation was transgranular. Further, multiple initiation sites were identified in most tests.

(4) Fatigue life of the coating was five times greater than that of coated PWA 1480 [001] single crystals.

(5) The high temperature fatigue life of the NiCoCrAlY was significantly greater than that at 650 °C and this difference was attributed to a slower transgranular crack growth rate at 1050 °C.

REFERENCES


TABLE I. - TENSILE PROPERTIES OF THE
NICoCrAlY ALLOY

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>650</th>
<th>1050</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2 percent yield strength, MPa</td>
<td>490</td>
<td>&lt;10&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Ultimate tensile strength, MPa</td>
<td>600</td>
<td>30</td>
</tr>
<tr>
<td>Elongation, percent</td>
<td>17</td>
<td>250</td>
</tr>
</tbody>
</table>

<sup>a</sup> $\dot{\varepsilon} = 7 \times 10^{-4}$ sec$^{-1}$. 

Figure 1. Miniature low cycle fatigue specimen.

Figure 2. Microstructure of the NiCoCrAlY alloy.
Figure 3. - Typical hysteresis loops.

Figure 4. - Cyclic softening observed at 650 °C.

Figure 5. - Cyclic stress-strain response at 650 °C.

Figure 6. - Fatigue life of the NiCoCrAlY compared to that of coated PWA 1480 at 650 °C.

Figure 7. - Elastic and inelastic life lines at 650 °C.

Figure 8. - Cyclic hardening observed at 1050 °C.
Figure 9. - Fatigue life of the NiCoCrAlY compared to that of coated PWA 1480 at 1050°C.

Figure 10. - Cyclic stress-strain response at 1050°C.

Figure 11. - Fatigue life of the NiCoCrAlY at 650 and 1050°C.
Figure 12. - Grain growth at 1050 °C as a function of time and applied strain.
Figure 13. - Surface damage was extensive at 1050 °C and minimal at 650 °C.
Figure 14. - Fracture initiation (top) and propagation (bottom) at 650 °C.

Figure 15. - Multiple surface initiation sites were observed at 1050 °C.
Figure 16. - Fracture initiation and propagation at 1050 °C.

Figure 17. - NiCoCrAIY strength was partially recovered by short time anneals at 760 °C.
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Fatigue; Superalloy; Coatings

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