High Cycle Fatigue Behavior of Polycrystalline NiAl-0.28 mol%Fe

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The high cycle fatigue (HCF) behavior of NiAl-0.28Fe (Ni-50.3 mol%Al-0.28 mol%Fe) was determined at four temperatures between 673 K and 928 K, below and above its ductile to brittle transition temperature, DBTT, (≈730 K) obtained from tensile tests. Fracture and surface slip band-characteristics were examined using an optical microscope and a scanning electron microscope. The fatigue resistance of NiAl-0.28Fe decreased significantly with increasing temperature, due to a creep-fatigue interaction above the DBTT. Below the DBTT cracks initiated at or near the surface with fatigue zones and overload zones consisted of a mixture of intergranular and transgranular fracture paths. Above the DBTT crack initiation occurred internally at or near microvoids (without observed fatigue zones), leading to transgranular fracture paths. Cross slip and wavy slip were exhibited in tensile and HCF specimens below and above the DBTT. NiAl-0.28Fe displayed higher fatigue resistance than Astroloy (powder metallurgy nickel-based superalloy) and Ni3Al+B in the range 673 K to 823 K.

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Keywords: high cycle fatigue, NiAl-0.28Fe, cyclic deformation, NiAl, intermetallic compound, ductile to brittle transition temperature

I. Introduction

The potential of current conventional materials for high temperature use has been exploited to a large extent. For example nickel-based superalloys have reached their upper temperature limit of application. The aerospace industry is therefore looking forward to the development of alternative materials that are lighter and stronger and have higher temperature potential compared with presently available alloys. In response to the need for structural materials, ordered intermetallic alloys have been the subject of intensified investigation for the past decade. Of the many intermetallic alloys under consideration, the B2 intermetallic compound NiAl and its alloys are one of the few systems that has emerged as a promising candidate for further development, due to such desirable properties as low density, high melting point and excellent oxidation resistance(6). However, poor ductility at room temperature has been one of the barriers limiting its practical application.

The low-temperature ductility of some intermetallics can be improved by techniques such as micro- and macro-alloying with small and large additions of ternary alloying elements, grain size refinement, martensite transformation and reinforcement by ductile fibers or particles. Darolia et al.(3) indicated recently that in room temperature tensile tests, up to 6% plastic elongation to failure was obtained in ⟨110⟩ oriented NiAl single crystalline specimens containing 0.1 mol%Fe and 0.25 mol%Fe. Limited low cycle fatigue data of NiAl have been published in the open literature(3-9). High cycle fatigue (HCF) of NiAl is an even more recent area of nickel aluminide research. The purpose of this paper is to determine HCF properties, and fracture and surface slip band-characteristics of a microalloyed (Fe=0.28 mol%) polycrystalline NiAl alloy at several temperatures (673–928 K), below and above the ductile to brittle transition temperature (DBTT) obtained from tensile tests, and to compare the HCF behaviors of NiAl-0.28Fe with those of high-temperature materials such as Ni3Al and nickel-based superalloys.

II. Experimental Procedures

A vacuum-induction melted ingot of NiAl-Fe was obtained from the General Electric Co. The ingot was canned in mild steel and was hot-extruded at 1173 K using an 8:1 reduction ratio at Oak Ridge National Lab. After extrusion, the canning material was removed by wire electro-discharge machining (EDM). Table 1 shows the chemical composition of the NiAl–Fe material used in this investigation. Compositions are given in mole percent.

The extruded rod was homogenized at 1373 K for 3.6 ks in a low purity argon stream in order to prevent intergranular disintegration upon oxidation(26). Button-head type tensile and HCF specimens with gauge diameters of 3.2 mm and gauge lengths of 19 mm were produced by low stress grinding, with the longitudinal directions of specimens parallel to the extruded direction. The speci-

<table>
<thead>
<tr>
<th>Table 1</th>
<th>The chemical composition of the as-extruded material (mol%).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>49.4</td>
</tr>
<tr>
<td>Al</td>
<td>50.3</td>
</tr>
<tr>
<td>Fe</td>
<td>0.28</td>
</tr>
<tr>
<td>O</td>
<td>0.02</td>
</tr>
</tbody>
</table>

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mens were annealed at 773 K for 3.6 ks in argon to remove residual grinding strains. They were mechanically polished and then electropolished in a solution of 10% perchloric acid, 45% methanol and 45% ethylene glycol at 233 K. Tensile tests were conducted at seven temperatures between 300 K and 928 K under an initial strain rate of $4.4 \times 10^{-4}$/s in air and were reported on previously(9).

The HCF tests were performed with a constant load amplitude using a sinusoidal waveform with a frequency of 20 Hz at four temperatures of 673, 823, 873 and 928 K; these are below and above the DBTT ($\pm 730$ K) obtained from tensile tests. All tests were performed in tension-tension loading in air with a constant minimum stress of 34.5 MPa and with varying maximum stresses of 109 MPa–180 MPa. The normalized stress values ($\sigma_{\text{Max}} / \sigma_{\text{YS}}$) obtained by dividing the maximum stress, $\sigma_{\text{Max}}$, by the 0.2% offset yield stress, $\sigma_{\text{YS}}$, were from 1.5 to 2.1.

After both tensile and HCF tests, the fracture surfaces and surface slip bands were observed using an optical microscope and a scanning electron microscope (SEM). Foils for transmission electron microscopy (TEM) were prepared from the cross section of the gauge length of a fractured tensile specimen by jet polishing at 233 K, in order to examine the dislocation structures. The electrolyte used was a solution of 60% methanol, 36% 2-butoxyethanol and 4% perchloric acid and polishing was carried out at 20 V and 8–10 mA. The foils were observed in a JEOL 100CX transmission electron microscope operating at 100 kV.

### III. Results and Discussion

1. **Microstructural characterization**

The microstructures of transverse and longitudinal sections for the as-extruded material were presented in earlier work(9). The average linear intercept grain size, $d$, was 29 $\mu$m.

Figure 1 shows the microstructures of transverse and longitudinal sections after homogenizing heat-treatment at 1373 K for 3.6 ks in argon. The average linear intercept grain size, $d$, was 130 $\mu$m, indicating that grain growth occurred after recrystallization.

2. **Tensile properties**

Tensile tests were carried out on NiAl–0.28Fe specimens ($d=130 \mu$m) at seven temperatures: 300, 589, 673, 723, 743, 824 and 928 K, and on a specimen ($d=29 \mu$m) at 300 K, and were reported on earlier(9). These data established the DBTT and base-line data from which cyclic testing temperatures and stress levels could be selected. The DBTT, defined as the temperature at which plastic strain to failure, $\varepsilon_p=5\%$, was established at approximately 730 K(9).

3. **HCF properties**

HCF tests were conducted on NiAl–0.28Fe with $d=130 \mu$m at four temperatures: 673, 823, 873 and 928 K; below and above the DBTT ($\pm 730$ K). The results of the HCF tests are listed in Table 2 and plotted as a function of temperature in Fig. 2. Each point represents a single test; arrows indicate test termination prior to failure. The fatigue resistance (measured as the number of cycles to failure, $N_t$) of NiAl–0.28Fe decreased markedly with increasing temperature. At 673 K and 823 K the difference in applied maximum stress at the fatigue limit ($10^7$ cycles) was approximately 54%. At an applied maximum stress of 109 MPa, there were about six- and three hundred-fold increases in fatigue life as temperature decreased from 928 K to 873 K and from 928 K to 823 K, respectively. The fatigue resistance among the different temperatures was compared in $\sigma_{\text{UTS}}$ and $\sigma_{\text{YS}}$-normalized plots, (see Figs. 3 and 4) where normalized data are obtained by dividing $\sigma_{\text{Max}}$ by the respective ultimate tensile strength, $\sigma_{\text{UTS}}$, and $\sigma_{\text{YS}}$. The $\sigma_{\text{UTS}}$-normalized plot indicates that the resistance to fatigue fracture decreases with increasing temperature. At 673 K the long fatigue life ($=1.93 \times 10^6$ cycles) was shown in the $\sigma_{\text{UTS}}$-normalized value of 0.99, which is extremely close to the $\sigma_{\text{UTS}}$. The
Table 2. The result for NiAl-0.28 mol%Fe with $d=130 \mu m$ tested under load-controlled high cycle fatigue with a constant minimum stress of 34.5 MPa at frequency of 20 Hz in air.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Number of cycles to failure, $N_f$</th>
<th>Reduction of area after test (%)</th>
<th>Elongation after test (%)</th>
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<tr>
<td>Temperature, $T/K$</td>
<td>$\sigma_{\text{max}}/\text{MPa}$</td>
<td></td>
<td></td>
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<tr>
<td>673</td>
<td>180</td>
<td>$1.93 \times 10^8$</td>
<td>25.3</td>
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<td>673</td>
<td>172</td>
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<td>14.4</td>
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<td>673</td>
<td>168</td>
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<td>11.2</td>
</tr>
<tr>
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<td>154</td>
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<td>13.0</td>
</tr>
<tr>
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<td>1.7</td>
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<tr>
<td>823</td>
<td>127</td>
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<td>54.7</td>
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<td>111</td>
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<td>48.2</td>
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<tr>
<td>823</td>
<td>109</td>
<td>$9.14 \times 10^3$</td>
<td>47.8</td>
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<tr>
<td>873</td>
<td>109</td>
<td>$1.76 \times 10^3$</td>
<td>62.3</td>
</tr>
<tr>
<td>928</td>
<td>109</td>
<td>$2.96 \times 10^3$</td>
<td>65.5</td>
</tr>
</tbody>
</table>

* Test termination prior to failure

Fig. 2 Effect of temperature on fatigue lives for NiAl-0.28 mol%Fe with $d=130 \mu m$ tested under load-controlled high cycle fatigue with a constant minimum stress of 34.5 MPa at frequency of 20 Hz in air. Arrows indicate test termination without failure.

Fig. 3 The data normalized by $\sigma_{\text{UTS}}$ for NiAl-0.28 mol%Fe with $d=130 \mu m$ tested under load-controlled high cycle fatigue with a constant minimum stress of 34.5 MPa at frequency of 20 Hz in air. Arrows indicate test termination without failure.

Fig. 4 The data normalized by $\sigma_{\text{UTS}}$ for NiAl-0.28 mol%Fe with $d=130 \mu m$ tested under load-controlled high cycle fatigue with a constant minimum stress of 34.5 MPa at frequency of 20 Hz in air. Arrows indicate test termination without failure.

test temperatures.

4. Fracture morphologies in HCF specimens

The fracture surfaces were relatively flat and no macroscopic necking occurred in the gauge section in the specimens tested at 673 K, see Fig. 5(a). There was macroscopic necking relating to localized deformation adjacent to the fracture surface in the specimens tested at 823 K and above, see Fig. 5(b).

Specimens tested at 673 K exhibited crack initiation at or near the surface [see, Fig. 5(a)] and the fracture morphology showed fatigue zones with fatigue striations, similar to a B2-type alloy (Fe-28.6Al-4.8Cr-0.21C-0.5Nb) tested at 300 K in air previously in our laboratory\(^6\). These specimens also showed overload zones consisting of a mixture of intergranular and transgranular fracture paths, similar to the tensile specimens below the DBTT\(^7\). No indication of a defined fatigue zone was observed for specimens tested at temperatures from 823 K to 928 K. Cracks appeared to initiate internally at or near microvoids, leading to transgranular fracture paths, such as that exhibited in Fig. 6.
5. Optical observation of surface slip bands

Below and above the DBTT surface slip bands were investigated on both tensile and HCF specimens with \( d = 130 \mu m \), shown in Figs. 7 and 8. Both cross slip and wavy slip were exhibited in each specimen, similar to a Ni–50Al polycrystal. The slip bands consisted of independent slip systems and cracking was observed along the slip bands. The bands were substantially indetical in all tests, supporting the idea that slip bands in HCF specimens resemble slip bands during Stage 3 hardening in tensile tests.

Wavy slip shows continuity of the slip bands across grain boundaries, which suggests that they are the result of cross slip rather than merely the motion of independent dislocations on intersecting slip planes. For NiAl-0.28Fe the slope \( k_s \) in the Hall-Petch relation:

\[
\sigma_s = \sigma_0 + k_s d^{-1/2}
\]

was 0.286 MPa (m)^{1/2} in the range of \( d = 29 \mu m - 130 \mu m \) at 300 K. The intercept \( \sigma_0 \) represents the lattice resistance to plastic flow. Since \( k_s \) is a measure of the strengthening due to dislocation/grain boundary interactions, the presence of cross slip in NiAl-0.28Fe accounts for a low value of \( k_s \). Since dislocation pile-ups do not readily occur, the grain boundaries do not experience a high localized stress. The presence of cross slip also suggests a high stacking fault energy.

6. TEM observation of a tensile specimen

The as-extruded specimen \( (d = 29 \mu m, \epsilon_p = 0.6\%) \) tested in tension at 300 K was examined by TEM. The TEM micrographs exhibits single and tangled dislocations, and some dislocation dipoles and loops, shown in Fig. 9. The major part of specimens; however, shows mainly single dislocations, in Fig. 9(a). The dislocation structures of the NiAl-0.28Fe polycrystal are similar to those of a Ni–50Al single crystal tested in tension at 300 K, but the density of dislocation dipoles in NiAl-0.28Fe appears lower than that of a Ni–50Al single crystal. At 300 K the dislocation density of the NiAl-0.28Fe polycrystal is much higher.
lower than that\(^{(2)}\) of a Ni–50Al polycrystal strained to approximately 2% at a strain rate of \(1 \times 10^{-4}\)/s and a \(\langle 110 \rangle\) oriented NiAl–0.25Fe (Ni–49.75Al–0.25Fe) single crystal showing \(\varepsilon_p\) of 6.6% at a strain rate of 8.3 \(\times 10^{-3}\)/s. The low dislocation density of the NiAl–0.28Fe polycrystal found here is consistent with the low plastic strain (\(\varepsilon_p\) = 0.6%).

The similar dislocation structures are found in each specimen of Ni–50Al which is monotonically tensile and cyclically deformed at room temperature, especially with respect to a zig-zag morphology\(^{(6)}\). The dislocation structures found in Ni–50Al after room temperature-cyclic deformation may be predictable from those of tensile deformed Ni–50Al. As mentioned earlier, for room temperature-tensile deformed specimens the dislocation structures of Ni–50Al are similar to those of NiAl–0.28Fe. On the other hand, the same surface slip band-characteristics were shown in both tensile and HCF specimens, see Figs. 7 and 8. Therefore, it may be also suggested that dislocation structures found in HCF specimens of NiAl–0.28Fe below DBTT are almost similar to those found in the tensile deformed NiAl–0.28Fe (Fig. 9) or Ni–50Al.

7. Fatigue-creep interaction

The most notable result of the HCF tests at elevated temperatures is the rapid drop in fatigue life above the DBTT. Figure 10 shows the temperature dependence of HCF lives and ductility, measured as the reduction of area and as the total elongation of the gauge length after HCF tests with a constant minimum stress of 34.5 MPa at a constant \(\sigma_{YS}\)-normalized ratio (\(\sigma_{MAX}/\sigma_{YS}\) = 1.8). The data show a significant decrease in number of cycles to failure, \(N_f\), and significant increases in the reduction of area and the total elongation, with increasing temperature. This result somewhat resembled the dependence of tensile properties such as \(\sigma_{UTS}\), \(\sigma_{YS}\) and \(\varepsilon_p\) on temperature\(^{(7)}\). Accompanying this sharp drop in fatigue lives was a large increase in localized deformation adjacent to the fracture surface, see Figs. 5(b) and 6(a). This clearly indicates evidence for the role of creep in the fatigue process above the DBTT. The presence of internal microvoids near the initiation site on the fracture surface at 928 K supports the role of creep in the failure process, Fig. 6(b).
Fig. 9 TEM micrographs shown in a tensile specimen of NiAl-0.28 mol%Fe produced from the as-extruded material with \( d = 29 \mu \text{m} \) showing \( \epsilon_r \) of 0.6\% at 300 K. Single dislocations (S) and dislocation dipoles (D) in (a). Tangled dislocations and dislocation loops (arrow) in (b).

Fig. 10 Effect of temperature on number of cycles to failure, \( N_f \), and ductility, measured as the reduction of area and as the total elongation of the gauge length after high cycle fatigue tests with a constant minimum stress of 34.5 MPa at a constant \( \sigma_{YS}\)-normalized ratio \( (\sigma_{YS}/\sigma_{YS}) = 1.8 \). An arrow indicates a test termination without failure.

8. Comparisons with other alloys

It is interesting to compare the HCF behavior of NiAl-0.28Fe with that of other high-temperature materials such as Ni$_3$Al and Ni-base superalloys. Figure 11 shows a \( \sigma_{YS}\)-normalized plot of HCF behavior of NiAl-0.28Fe and Astroloy\textsuperscript{[25]} and Ni$_3$Al+B (Ni-23.82Al-1.25Fe-0.21B)\textsuperscript{[26]} tested previously in our laboratory. NiAl-0.28Fe displays the highest fatigue resistance of the three alloys tested in the range of 673 and 823 K. At 823 K the endurance ratio (\( \Delta \sigma_{OP}/\sigma_{YS} \)) of NiAl-0.28Fe is 1.19 which is from 1.3- to 2.0-times higher than those (0.60 and 0.92) of Astroloy with fine \( \gamma^\prime \) (0.08 \( \mu \text{m} \)) and coarse \( \gamma^\prime \) (0.8-1.2 \( \mu \text{m} \)) particles, respectively, although exact comparison is difficult because of the different test atmospheres. Fatigue resistance of NiAl-0.28Fe is similar to that of Astroloy with coarse \( \gamma^\prime \) particles in the range of 923-928 K.

IV. Conclusions

1. The fatigue resistance of NiAl-0.28Fe decreased significantly with increasing temperature, due to a creep-fatigue interaction above (823–928 K) the DBTT.

2. HCF specimens tested at 673 K (below the DBTT) exhibited crack initiation at or near the surface. The fracture morphology showed fatigue zones and overload zones consisted of a mixture of intergranular and transgranular fracture paths. For HCF specimens tested above (823 K–928 K) the DBTT, crack initiation occurred internally at or near microvoids (without observed fatigue zones), leading to transgranular fracture paths.

3. Cross slip and wavy slip were exhibited in both tensile and HCF specimens below and above (673 K and 823 K or 824 K) the DBTT.

4. NiAl-0.28Fe displayed higher fatigue resistance than Astroloy (nickel based superalloy) and Ni$_3$Al+B in the range of 673 K–823 K.

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