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THE TEMPERATURE AND STRAIN RATE DEPENDENCE OF THE FLOW STRESS OF SINGLE CRYSTAL NiAl DEFORMED ALONG $\langle 110 \rangle$

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Abstract

Single crystal NiAl and Ni-49.75Al-0.25Fe have been deformed along $\langle 110 \rangle$ at temperatures of 77, 298 and 773K and strain rates of 0.001/s, 0.1/s and 2000/s. The flow stress of $\langle 110 \rangle$ NiAl is rate and temperature sensitive. The 0.25 at.% Fe addition resulted in a small increase in flow stress at strain rates of 0.001 and 0.1/s at 298 and 77K. A significant decrease in the work hardening rate is observed after deformation at 77K and a strain rate of 2000/s. Coarse $\{110\}$ slip traces are observed after deformation at a strain rate of 2000/s at 77K, while no slip traces were observed after deformation under all other conditions. TEM observations reveal distinct $\{110\}$ slip bands after deformation at 77K and a strain rate of 2000/s.

Introduction

NiAl has been studied previously as a potential high temperature structural material because of its many attractive properties such as low density, high temperature oxidation resistance, and good ductility at room temperature. A review of the research literature on NiAl has been presented by Miracle [1] and Noebe, Bowman, and Nathal [2]. This work shows that among other things, the properties of NiAl are quite anisotropic. For example, the yield strength along $\langle 100 \rangle$ at room temperature is ~ 1500 MPa while that along $\langle 110 \rangle$ is 1/10th of that value (150 MPa).

The mechanical properties of single crystal NiAl along $\langle 110 \rangle$ have been studied by many authors [3-7]. Slip has been confirmed through careful TEM investigations to be on the $\{110\}\langle 001 \rangle$ and $\{100\}\langle 001 \rangle$ slip systems. In depth studies of deformation at room temperature have shown that the CRSS for the $\{100\}\langle 001 \rangle$ and $\{110\}\langle 001 \rangle$ slip systems are similar but slightly lower for the former slip system [3,5]. Pascoe and Newey [6] note that for deformation at 77K, only $\{110\}\langle 001 \rangle$ slip is observed.

Point defects and impurities can have a strong influence on the mechanical properties of single crystal NiAl along $\langle 110 \rangle$. Additions of small amounts (less than 0.5 at.%) of Fe, Mo and Ga can improve the tensile ductility along $\langle 110 \rangle$ to as much as 6% at room temperature [8]. The mechanism by which the Fe, Mo, and Ga additions enhance the room temperature ductility is not known although it may be an indirect effect of point defect or impurity gettering by the small additions [9]. Improvements in tensile ductility to as much as 5% along $[123]$ have been observed in very high purity single crystals (C, N, and S each less than 5 wppm and O around 10-16 wppm) [10].

The rate sensitivity of single crystal NiAl along $\langle 111 \rangle$, $\langle 112 \rangle$, and $\langle 123 \rangle$ (a plastic instability occurred along $\langle 110 \rangle$) has been studied at temperatures from 473 to 873°C in tension by means of strain rate jump tests from 1.25×10^{-4} /s to 1.25×10^{-3} /s [7]. The activation volume ranged from 2000 to 5000 b^3 . The rate sensitivity of single crystal NiAl along $\langle 110 \rangle$ was also studied by Lahrman, Field and Darolia [11] in tension at strain rates of 8×10^{-3} /s and 8×10^{-5} /s. No effect of strain rate on the ductile-to-brittle temperature (DBTT) was observed. On the other hand, a significant effect of strain rate on the DBTT in polycrystalline NiAl in tension has been observed. As the strain rate was increased from 1.4×10^{-4} /s to 1.4×10^{-1} /s, the DBTT increased by 200°C [12]. In recent work by Kitano and Pollock [13], the rate sensitivity of single crystal NiAl along $[110]$ with and without 0.25 at.% Fe additions was studied. Rate jump tests were performed in compression at temperatures from RT to 873K by jumping the strain rate from 1.75×10^{-4} /s to 1.75×10^{-2} /s. In NiAl with and without Fe additions, the rate sensitivity was fairly constant with temperature and ranged from 0.026 to 0.012. The activation volume was also calculated as $60b^3$ at RT and increased to 400-500 b^3 at 873K. The large activation volumes at room temperature and above suggest that flow of $\langle 100 \rangle$ dislocations is not controlled by a large Peierls barrier at these temperatures.

Although much work has been performed on the mechanical properties of NiAl along $\langle 110 \rangle$ at strain rates ranging from $10^{-5}/s$ to $10^{-1}/s$, no work has been performed at higher strain rates. In this study, the temperature and strain rate dependence of the flow stress of single crystal NiAl deformed in compression along $\langle 110 \rangle$ is investigated through compression testing at strain rates of 2000/s, 0.1/s and 0.001/s and temperatures of 77K, 298K and 773K. Some initial results of the slip traces and TEM analyses are reported to relate the mechanical properties to the active slip systems responsible for flow in this material.

Experimental Procedure

Single crystals of stoichiometric NiAl and Ni-49.75Al-0.25Fe were grown by the Bridgman technique at General Electric Aircraft Engines. The single crystals were homogenized at 1588K for 50 hours in argon followed by furnace cooling. The crystals were then oriented along $\langle 110 \rangle$ using the Laue back-reflection X-ray technique and cylindrical specimens (5mm diameter x 5mm height) were EDM wire cut from them. Following this, the specimens were chemically polished in a solution of 85% phosphoric acid and 15% nitric acid at 343K for 25 minutes to remove any EDM damage and polish the surfaces.

Quasi-static compression tests at strain rates of 0.001 and 0.1/s were performed on a screw-driven Instron testing machine using a molybdenum grease lubricant at 77K and 298K. Dynamic tests at a strain rate of $\sim 2000/s$ were conducted at 77K, 298K and 773K using a Split-Hopkinson pressure bar (SHPB). The oscillations in the SHPB stress/strain curve reflect real elastic wave dispersion in the data and accurate stress data at low strains is not possible until the stress in the sample is stable; accordingly, it is difficult to accurately assess the yield strength. A vacuum furnace was used for tests performed at 773K. At least two tests were performed for each condition to confirm each result. Samples tested at elevated temperatures were lubricated with boron nitride prior to testing [14].

After testing, slip traces were observed using a scanning electron microscope (SEM) operated at 25 kV. Following mechanical testing, the samples were cut parallel to the slip plane. If no slip plane was evident, the samples were sectioned parallel to the loading direction. Foils for transmission electron microscopy (TEM) were prepared by mechanically thinning the sectioned sample slices to 150 μm , and electropolishing with a Struers Tenupol using a solution of 7% perchloric acid and ethanol at 246K. TEM foils were observed using a Philips CM30 Transmission Electron Microscope operating at 300KV.

Results

Mechanical Properties

The stress/strain curves for compression testing of single crystal NiAl and Ni-49.75Al-0.25Fe at strain rates of 0.001 and 0.1/s and 298 and 77K are shown in Fig. 1. The stage II work hardening rates (Table I) are the highest for deformation at a strain rate of 0.001/s at 77K. Normalizing the work hardening rate, θ , with the Taylor Factor, M , for a random BCC polycrystal since data for NiAl is not known, $[\theta/3.06^2]$, gives a hardening rate of $\mu/84$ at a strain rate of 0.001/s at 77K. (The shear modulus along $[110]$ is 52.5 GPa [1].) The addition of 0.25 at.% Fe resulted in a slight increase in the yield stress with the stress/strain curves overlapping each other after about 8% strain. This increase was confirmed with a second test in each condition although more tests are needed to statistically confirm this increase in yield stress with Fe content.

The stress/strain curves for compression testing of single crystal NiAl at 2000/s and temperatures of 77K, RT, and 773K are shown in Fig. 2. No differences were observed with the 0.25 at.% Fe addition so this data is not shown. Fig. 2 shows that single crystal NiAl is rate sensitive along $[110]$. A measure of the rate sensitivity (m) can be made from the yield stress at 2000/s and 0.001/s by the following formula [15]:

$$m = \ln(\sigma_{y1}/\sigma_{y2}) / (\ln(\dot{\epsilon}_1/\dot{\epsilon}_2)) \quad (1)$$

where σ_{y1} and σ_{y2} are the yield strengths at the strain rates, $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$. The accepted method for

Figure 1 Stress/Strain curves for single crystal NiAl and Ni-49.75Al-0.25Fe tested along $\langle 110 \rangle$ in compression at temperatures of 77K and RT and strain rates of 0.001/s and 0.1/s.

Figure 2 Stress/strain curves for deformation of single crystal NiAl along $\langle 110 \rangle$ in compression at temperatures of 77, RT and 773K and strain rates of 2000, 0.1 and 0.001/s.

Table I Selected Mechanical Properties for Compression Testing of Single Crystal NiAl along $\langle 110 \rangle$

Conditions (strain rate, Temperature)	0.2% yield stress (MPa)	Work Hardening Rate (MPa/unit strain)
0.001/s, 77K	828 878 (with 0.25% Fe)	5730
0.1/s, RT	198 213 (with 0.25% Fe)	3170
0.001/s, RT	160 173 (with 0.25% Fe)	2900
2000/s, 77K	~1300	1800
2000/s, RT	~500	3560
2000/s, 773K	~160	2620

measuring rate sensitivity is by performing a rate jump test in which the strain rate during deformation is jumped between two different strain rates and a constant dislocation substructure is held during the test. Using the yield stress from two separate tests, a rate sensitivity of 0.085 is observed at RT and a rate sensitivity of 0.028 is observed at 77K. Similar work hardening rates are observed at RT and 773K at 2000/s to those observed after deformation at a strain rate of 0.001/s (Table I), but the smallest work hardening rate is observed after deformation at 77K and a strain rate of 2000/s. Large yield drops of ~300 MPa are observed during deformation at RT and 773K. This observation was repeated in identical tests. It is unknown if these load drops reflect a BCC-type yield drop phenomenon or experimental manifestations of the SHPB stress state as it stabilizes at low strains. To test the validity of this result, a test is in progress in which the strain will be measured on the sample using strain gages.

Slip Trace Analysis

Slip traces were only observed after deformation at a strain rate of 2000/s at 77K. Slip traces were not observed after deformation under other testing conditions. Figure 3 shows an SEM micrograph of the slip traces on the (110) face after deformation at 2000/s and a temperature of 77K to a plastic strain of 10%. These slip traces are on the (101) and (011) planes. Many microcracks, most of which were perpendicular to the compression direction were also observed.

TEM

Figure 4 shows a TEM micrograph of a sample tested at a strain rate of 2000/s at 77K. Slip bands are observed containing a high density of dislocations. These slip bands are present on (101) and (011) planes consistent with the slip trace analysis. Further in depth TEM is in progress.

Discussion

The temperature and strain rate sensitivity of the flow stress of single crystal NiAl tested along $\langle 110 \rangle$ is shown in Fig. 2. At 77K, a rate sensitivity is observed but the most noticeable difference is the change in the work hardening rate. The work hardening rate decreases three orders of magnitude as the strain rate is changed from 0.001/s to 2000/s. This change is accompanied by the presence of distinct (101) and (011) slip traces and coarse slip bands are also observed using TEM. Planar slip allows for easy glide without a large number of dislocation interactions, thereby reducing the overall work-hardening rate.

A higher rate sensitivity is observed after deformation at RT. Also, the work hardening rate doubles over that observed at 77K and 2000/s. This may be a result of slip occurring on (100) and (010) planes (as has been reported previously for deformation at slower strain rates [3])

Figure 3 SEM micrograph showing $\{011\}$ slip traces on the (110) face of a $\langle 110 \rangle$ single crystal NiAl specimen tested at a strain rate of 2000/s at 77K (arrow shows compression direction).

Figure 4 TEM micrograph showing a (101) slip band after deformation at a strain rate of 2000/s at 77K.

resulting in more dislocation interactions.

In comparison with previous work, the observation of {110} slip after deformation at 77K and 2000/s is not too surprising. The {110} plane is the closest-packed plane in NiAl and slip on {110} planes has been reported previously after deformation at slower strain rates at 77K along $\langle 110 \rangle$ [6].

The yield stress for stoichiometric single crystal NiAl at room temperature along $\langle 110 \rangle$ is similar to those values observed by Kitano and Pollock [13] but smaller than the yield stress reported by other authors [3,7]. Our results suggest a slight increase in yield strength with Fe addition while the results of Darolia [10] suggest a decrease in yield stress with Fe addition. The yield stress of single crystal NiAl is strongly dependent on impurity content as well as heat treatment [10] which may explain these subtle differences.

Conclusions

Based upon a study of the influence of strain rate and temperature on the substructure evolution and stress-strain response of $\langle 110 \rangle$ NiAl, the following conclusions can be drawn:

- 1) The flow stress of $\langle 110 \rangle$ NiAl is rate and temperature sensitive; the rate sensitivity is 0.085 at 293K and 0.028 at 77K.
- 2) $\langle 110 \rangle$ NiAl exhibits a high and nearly linear work hardening behavior.
- 3) The work hardening rate is the lowest (1800 MPa/unit strain) after deformation at 77K and 2000/s.
- 4) TEM and slip trace analyses reveal intensely coarse {110} slip traces after deformation at 77K and a strain rate of 2000/s.
- 5) The addition of 0.25% Fe results in a small increase in flow stress at strain rates of 0.001/s and 0.1/s and temperatures of 77K and RT.

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