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High Strain Rate Deformation of NiAl

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Abstract

NiAl is a potential high temperature structural material. The applications for which NiAl is being considered (such as rotating components in jet engines) requires knowledge of its mechanical properties over a wide range of strain rates. Thus, single crystal NiAl (stoichiometric and Ni 49.75Al 0.25Fe) has been deformed in compression along [100] at strain rates of 0.001, 0.1/s and 2000/s and temperatures of 76, 298 and 773K. $\langle 111 \rangle$ slip was observed after 76K testing at a strain rate of 0.001/s and 298K testing at a strain rate of 2000/s. Kinking was observed after deformation at 298K and a strain rate of 0.001/s and sometimes at 298 K and a strain rate of 0.1/s. Strain hardening rates of 8200 and 4000 MPa were observed after 773K and 298K testing respectively, at a strain rate of 2000/s. The mechanical testing results are discussed in reference to the resulting dislocation substructure.

Introduction

NiAl is a promising intermetallic compound for high temperature structural applications owing to its high melting temperature, high strength, low density, high thermal conductivity and good oxidation resistance[1,2]. Although, NiAl suffers from low ductility at room temperature, microalloying with Fe, Ga, or Mn at levels less than 0.5% is known to increase the room temperature tensile ductility in the $\langle 110 \rangle$ direction to as much as 6%[3]. NiAl has the CsCl (B2) crystal structure. The shortest lattice translations are $\langle 100 \rangle$, 0.289 nm, $\langle 110 \rangle$, 0.409 nm and $\langle 111 \rangle$, 0.500 nm and the densest stacking plane is {110}. Accordingly, elementary dislocation theory would predict the easiest slip system to be {110} $\langle 001 \rangle$.

NiAl has been the subject of considerable experimental research. A summary of this work was recently provided by Miracle[2] and Noebe et al.[1]. For deformation in the soft orientation (e.g. $\langle 110 \rangle$) slip occurs by means of $\langle 100 \rangle$ Burgers vectors. For deformation in the hard orientation, (e.g. $\langle 100 \rangle$), $\langle 100 \rangle$ slip is not allowed. Thus, slip occurs by means of $\langle 111 \rangle$ dislocations at RT and 77K. Slip trace analyses at 77K reveal $\{112\}$ and $\{110\}$ slip planes. On the other hand, slip traces were indefinite after slip at RT and kinking was often observed which involves slip by means of $\langle 100 \rangle$ Burgers vectors[4,5]. Above the quasi-static DBTT which is between 623 and 673K at a strain rate of $10^{-4}/s$ in $\langle 100 \rangle$ oriented crystals, $\langle 110 \rangle$ dislocations become active[6].

The variation of yield stress with temperature has been measured in compression for stoichiometric single crystal NiAl. A gradual decrease in stress is observed for $\langle 111 \rangle$ slip as the temperature increases from 77K to 600K (1900MPa at 77K to 1250MPa at 600K). A sharp decrease in stress is observed above 600K (1250 MPa at 600K to 150MPa at 1000K) corresponding to the onset of $\{110\} \langle 110 \rangle$ slip[7,8].

Strain rate jump tests on single crystal NiAl have been performed at temperatures between RT and 873K[9]. From their data, activation volumes for $\langle 111 \rangle$ and $\langle 110 \rangle$ slip were calculated. The values of activation volume were low at all temperatures (3 for $\langle 111 \rangle$ slip at RT and 14-15 for $\langle 110 \rangle$ slip at 873K). Such a low activation volume suggests deformation by either $\langle 111 \rangle$ or $\langle 110 \rangle$ dislocations is highly restricted by either a large Peierls barrier or a complex dislocation core structure.

The effect of strain rate on the mechanical properties of polycrystalline NiAl at temperatures ranging from 500 to 1350K has received more limited attention. This work has shown that the largest effect of strain rate on yield strength is observed at temperatures above 750K. The strain rates were between 10^{-1} and $10^{-8}/s$ [10].

The aim of this work is to investigate the mechanical properties and substructure evolution of single crystal NiAl in compression along $[100]$ at strain rates up to 2000/s and

temperatures of 77, 298 and 773 K. This work will shed light on the behavior of single crystal nickel aluminide at high strain rates and plastic strains of up to 20%.

Experimental Procedure

Single crystals of stoichiometric NiAl and 50Ni49.75Al0.25Fe 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 [100] 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 surface.

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. Dynamic tests at a strain rate of ~2000/s were conducted at 298K and 773K using a Split-Hopkinson pressure bar. A vacuum furnace was used for tests performed at 773K. Samples were lubricated with boron nitride prior to testing[11].

After testing, slip traces were observed using a scanning electron microscope (SEM) operated at 25 kV. Following mechanical testing, the compression samples were sectioned parallel to the loading direction or if a slip plane was evident, it was sectioned. Foils for transmission electron microscopy (TEM) were prepared by mechanically thinning to 150 μm the sectioned sample slices and electropolishing with a Struers Tenupol using a solution of 7% perchloric acid and methanol at -40°C. Burgers vectors and line directions of dislocations were determined by performing $\mathbf{g \cdot b}$ and slip trace analyses using a Philips CM30 Transmission Electron Microscope operating at 300KV.

Results

The stress/strain curves obtained from quasi-static and dynamic testing of single crystal NiAl along [100] are shown in Fig. 1. These tests are representative of a minimum of two tests performed at each strain rate and temperature. The results shown are for stoichiometric NiAl although similar results were obtained for Ni_{49.75}Al_{0.25}Fe.

The strong influence of strain rate and temperature on the flow behavior in NiAl is clearly evident in Fig. 1. For deformation at 0.001/s, the yield stress decreases from 1800 to 1380 MPa as the temperature is increased from 77K to 298K. For deformation at room temperature, the stress at 3% strain increases from 1450 at a strain rate of 10⁻³ to 1750 at a strain rate of 2000/s (the stress at 3% strain is chosen for comparison to avoid the oscillations in the curve arising from elastic wave dispersion in the pressure bars). The rate sensitivity, m , may be measured from the change in yield stress with strain rate at room temperature. In order to assume a constant dislocation microstructure during testing, a rate jump test is usually performed. Thus, our calculations are only made to compare with previous calculations with rate jump tests. The rate sensitivity is measured from the following formula:

$$m = d \ln \dot{\epsilon} / d \ln \sigma$$

where $\dot{\epsilon}$ is the strain rate. Using the yield stress at 0.1/s and 0.001/s a rate sensitivity of 41 is calculated which is similar to the previously reported values of 53 and 65[9].

A difference in work hardening rates can be noted in Fig. 1. The 76K test performed at a strain rate of 10⁻³/s has a similar work hardening rate to the room temperature tests performed at 10⁻³/s, 0.1 and 2000/s. An increase in the work hardening rate with strain is observed for the RT tests at 10⁻³/s and 0.1/s (i.e. the stress/strain curves are concave up) This behavior is different from the behavior of disordered metals for which the work hardening rate decreases with strain[13,14,15]. In addition, the work hardening rates for deformation at strain rates of .001 and 2000/s at room temperature and a strain rate of 0.001 at 77K are approximately equal at 4000MPa ($\mu/28$ where $\mu=112$ GPa along [100] at RT[12]) while the work hardening rate rises to 8200 MPa ($\mu/14$ using the

same RT modulus; actually somewhat larger if the μ at 773K were used) at a strain rate of 2000/s at 773K.

For the tests performed at 298K and a strain rate of 0.001/s, kinking always occurs coincidently with a load drop in the stress/strain curve. For the tests performed at 0.1/s and 298K, kinking was sometimes observed accompanied by a load drop at 1400MPa. The 0.1/s stress/strain curve at 298K shown in Fig.1 is for a test in which no kinking was observed and smooth yielding occurred at 1550 MPa.

Slip traces observed on samples (compressed to ~2% plastic strain) tested at a strain rate of 0.001/s at 76 and 298K are shown in Figs. 2a+b. The slip traces on the sample tested at 76K correspond to $\{112\}$ slip with some cross-slip on $\{110\}$. The traces observed on the sample tested at 298K and a strain rate of 0.001/s result from kinking. One or two cracks were always observed associated with each kink.

For the samples tested at 298K and a strain rate of 2000/s, post-testing examination revealed that the specimens were cracked, but the in tact pieces exhibited coarse slip bands. No slip traces were found on the samples tested at 773K and a strain rate of 2000/s, and the samples exhibited no external macro-cracking after testing. This is interesting since 14% plastic strain was obtained at a strain rate of 2000/s and 773K (most probably below the BDTT at that strain rate[16]).

The dislocation substructure formed from deformation at a strain rate of 0.001/s at 76 and 298K are shown in Figs. 3a and b. G-b and trace analyses revealed that the dislocations had $\langle 111 \rangle$ Burgers vectors and were pure screw in character after deformation at 76K. Weak beam dark field imaging revealed that no dissociation was detected within the 2 nm resolution of the weak beam imaging technique. A very high density of dislocation tangles were observed after deformation 298K and a strain rate of 0.001/s. This sample had been plastically strained to 20%. Accordingly, it was not possible to perform a g.b analysis on the individual dislocations.

The dislocations formed from deformation at a strain rate of 2000/s at 298K and 773K are shown in Figs. 3c and d. The dislocations formed at 298K and a strain rate of 2000/s are $\langle 111 \rangle$ screw dislocations (Fig. 3c) as revealed by g·b and trace analyses. Dislocations formed from deformation at 773K and a strain rate of 2000/s are shown in Fig. 3d. A very high density of dislocations are present preventing a g.b analysis from being performed. Dislocations were curved and many tangles were observed.

Discussion

From the previously stated results, we can examine how NiAl deforms at temperatures of 76, 298 and 773K and strain rates of 0.001, 0.1 and 2000/s. For deformation at 76K, deformation occurs via the $\{112\} \langle 111 \rangle$ slip system. Slip is very planar and screw dislocations move slower than edge dislocations reminiscent of BCC metals[17,18]. Unlike BCC metals, the $\langle 111 \rangle$ dislocation is a superlattice dislocation which may dissociate into two $1/2 \langle 111 \rangle$ partials separated by an APB. This APB has a very high energy because its separation is less than 2 nm. Previous work supports these results[19].

At 298K, and a strain rate of 0.001/s, kinking was observed which is caused by massive movement of $\langle 001 \rangle$ dislocations[4,5]. Our observations of kinking are somewhat different than previous observations of kinking in NiAl. Previous observations have shown that kinking occurs in [100] NiAl single crystals at a strain rate of 1×10^{-4} /s at temperatures between 298 and 600K[1]. Increasing the strain rate at temperatures above 600K also promotes kinking[4,5]. For example, at 1050K, kinking is observed when the strain rate is increased to 10^{-2} /s. TEM investigations of the kinked regions reveal dislocations with $\langle 100 \rangle$ Burgers vectors and $\langle 110 \rangle$ Burgers vectors. Our observations show that at RT, when the strain rate is increased from 10^{-3} /s to 2000/s the deformation mode changes from kinking to uniform slip by $\langle 111 \rangle$ Burgers vectors.

Since kinking may be avoided by either decreasing the temperature to 77K or increasing the strain rate to 2000/s (at RT), this suggests that kinking is either thermally activated or diffusion controlled. It is highly unlikely that kinking is diffusion controlled because of the low diffusion rates at room temperature in NiAl and the short duration of the test performed at a strain rate of 10^{-3} /s. A more likely explanation is that glide occurs by $\langle 111 \rangle$ dislocations at room temperature. When deformed at a strain rate of 2000/s, the $\langle 111 \rangle$ dislocations glide as coupled $1/2\langle 111 \rangle$ partial dislocations with an APB between them. When deformed at a strain rate of 10^{-3} /s enough thermal energy and time is available for the $1/2\langle 111 \rangle$ partial dislocations to recombine and decompose into perfect $\langle 100 \rangle$ and $\langle 011 \rangle$ dislocations (this decomposition has previously been observed during deformation of [100] single crystals at 773K and a strain rate of 2×10^{-4} /s[20], and after room temperature deformation at a strain rate of 5×10^{-5} /s[17]). The $\langle 011 \rangle$ dislocations continue to glide leaving the $\langle 100 \rangle$ dislocations to form a kink band. Thus, at temperature above 600K, kinking would not be expected because glide occurs by $\langle 110 \rangle$ dislocations. Although, when the strain rate is increased at temperatures above 600K at low strain rates, the stress is high enough to nucleate $\langle 111 \rangle$ dislocations and kinking may occur.

At 773K, and a strain rate of 2000/s, deformation may be occurring by $\langle 111 \rangle$ dislocations which immediately decompose into $\langle 011 \rangle$ and $\langle 100 \rangle$ dislocations leaving the sample filled with a high density of dislocation tangles and loops. The resulting increase in storage of dislocation line length is accompanied by an increased work hardening rate[14,15].

Another way of looking at the stress/strain data is in terms of $\theta\sigma$ vs. σ (θ is the work hardening rate) where $\theta\sigma$ is a measure of the dislocation storage rate[14,15,21]. Fig. 4 shows a graph of $\sigma\theta$ vs. σ for the [100] NiAl samples in this study. From this graph, one can see that for tests performed at 2000/s, the dislocation storage rate is high, initially, and stays fairly constant for the duration of the test. Also, an increase in dislocation storage rate occurs as the temperature increases from RT to 773K at 2000/s. Finally, for

both the tests performed at RT and $10^{-3}/s$ and $10^{-1}/s$ strain rates, the dislocation storage rate is low initially and increases as the stress increases. An increase in the dislocation storage rate can be caused by either a decrease in the amount of cross-slip (such as in a test performed at 2000/s) or an increase in the density of forest dislocations (such as may occur from the decomposition of $\langle 111 \rangle$ dislocations).

Therefore, the different dislocation storage rates for the various tests can be described as follows. For 76K testing at a strain rate of $10^{-3}/s$ and room temperature testing at a strain rate of 2000/s, the work hardening rate is relatively constant or decreases gradually with strain. Deformation occurs by $\langle 111 \rangle$ slip and very little cross-slip occurs because of the low temperature or high strain rate conditions of the test. For room temperature testing at strain rate of 0.001/s and 0.1/s, the dislocation storage rates increase with strain because of the increase in the number of dislocation reactions and $\langle 111 \rangle$ dislocation decompositions into $\langle 100 \rangle$ and $\langle 011 \rangle$ dislocations which occur with continued plastic deformation. Finally, for the 773K tests performed at a strain rate of 2000/s, the stress is high enough to nucleate $\langle 111 \rangle$ dislocations although there is enough thermal energy for the $\langle 111 \rangle$ dislocations to immediately decompose resulting in a high initial dislocation storage rate which remains nominally constant with strain. In all tests, the dislocation storage rate is higher than observed for disordered metals because of the reduced dynamic recovery in intermetallics[21].

Summary

- 1) Single crystals of NiAl and Ni 49.75Al 0.25Fe have been deformed in compression along [100] at temperatures of 76, 298 and 773K and strain rates of 0.001, 0.1, and 2000/s.
- 2) G·b and trace analyses of the dislocation substructure revealed deformation by $\langle 111 \rangle$ dislocations at 77K and a strain rate of 0.001/s and 298K and a strain rate of 2000/s.
- 3) Kinking was observed after deformation at 298K and a strain rate of 0.001/s.

4) Kinking does not occur after deformation at 298K and a strain rate of 2000/s suggesting that kinking is caused by a thermally activated mechanism such as the decomposition of the $\langle 111 \rangle$ dislocation into $\langle 100 \rangle$ and $\langle 011 \rangle$ dislocations.

5) The work hardening rate was the highest, 8200 MPa, for deformation at 773K at a strain rate of 2000/s while the work hardening rate decreased to 4000 MPa ($\mu/28$) at 298K and a strain rate of 2000/s.

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Fig. Captions

Fig.1. Graph of true stress vs. true strain for mechanical testing of single crystal NiAl along [100] at temperatures of 76, 298, and 773K and strain rates of 0.001, 0.1 and 2000/s.

Fig. 2 SEM micrographs showing (a) {112} and {110} slip traces after 2% deformation at 76K and a strain rate of 0.001/s and (b) kink bands after 2% deformation at 298K and a strain rate of 0.001/s.

Fig. 3 (a) Weak beam dark field (WBDF) micrograph showing $\langle 111 \rangle$ screw dislocations after deformation at 76K and a strain rate of 0.001/s. (b) WBDF micrograph showing dislocation tangles after 20% plastic strain at 298K and a strain rate of 0.001/s. (c) Bright field (BF) micrograph showing $\langle 111 \rangle$ screw dislocations after deformation at 298K and a strain rate of 2000/s. (d) BF micrograph showing dislocation tangles and loops after deformation at 773K and a strain rate of 2000/s.

Fig. 4 Graph of $\theta\sigma$ (dislocation storage rate) vs. true stress for single crystal NiAl deformed along [100] at temperatures of 298 and 773K and strain rates of 0.001, 0.1 and 2000/s.

Fig.1. Graph of true stress vs. true strain for mechanical testing of single crystal NiAl along [100] at temperatures of 76, 298, and 773K and strain rates of 0.001, 0.1 and 2000/s.

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Fig. 3 a + b

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