Plastic Deformation by Slip on (001)[100] in Ni₃Nb Single Crystals with D₀₅ Structure

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Plastic deformation by slip on (001)[100] in Ni₃Nb single crystals was investigated. The yield stress, strain rate sensitivity of the flow stress, shape of the stress-strain curve, morphology of slip markings and deformation substructures varied strongly depending on temperature. Temperature dependence of their behavior was classified into three regions. Anomalous increase in the yield stress was observed between 500 and 800°C. The anomalous strengthening is caused by dynamic strain aging, the so-called Portevin-Le Chatelier effect.

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1. Introduction

An intermetallic compound Ni₃Nb with the D₀₅ structure has been of considerable interest as a strengthening phase of unidirectionally solidified eutectic alloys composed of Ni₃(Al)₇/Ni₃Al(γ′)-Ni₃Nb(σ) phases for high-temperature structural materials (for example, see references1,2). Recently, our group reported the deformation mode and the temperature dependence of yield stress of Ni₃Nb single crystals3−5). Four slip systems of (010)[100], (010)[001], (001)[100] and (201)[102], and three twinning systems of [111][10 7 13], [011][011] and [012][021] were operative depending on crystal orientation and temperature.3 Among their deformation modes, (010)[100] slip was dominant in low temperature range, and the critical resolved shear stress (CRSS) for (010)[100] slip displayed a positive temperature dependence. The anomalous strengthening is caused by the Kear-Wilsdorf (K-W) locking of a 1/2[100] superpartial dislocation cross-slipped from (010) to (001).2 Macroscopic [100] slip on (001) was also observed and the plastic behavior was examined in compression at low temperatures using [302]* oriented samples.5. Here, an asterisk(*) indicates the reciprocal space representation; [hkil]* is parallel to the (hkil) normal. The plastic deformation behavior of the (001)[100] slip could not be clearly understood because several types of deformation twin easily operated in compression at high temperatures3,4). The plastic behavior of the (001)[100] slip was examined at high temperatures by tensile test using [205]* oriented sample in which activation of twinning was suppressed.5)

In this study, the detailed deformation behavior by slip on (001)[100] in Ni₃Nb single crystals with the D₀₅ structure was examined in a wide temperature range focusing on the dynamic aging for forming dragging atmosphere around moving dislocations and the anomalous strengthening mechanism.

2. Experimental Procedure

Master ingot of a stoichiometric Ni–25.0 at%Nb was prepared by melting high purity Ni and Nb in a plasma arc fur- nace in high purity Ar gas. Single crystal was grown by the floating zone method using NEC SC-35HD furnace at a rate of 2.5 mmh⁻¹ under Ar gas flow. Plate-like samples with the gage dimensions of approximately 2 × 1 mm² × 5 mm were prepared for tensile test by the electro-discharge machining from the as-grown single crystal. Loading axis was chosen to be [205]*. For this orientation, Schmid factor (SF) for (001)[100] slip is large to be 0.315 and SFs for (010)[100] and (010)[001] slip are negligible. The detailed preparation method should be referred to the previous papers3,5).

Tensile tests were performed on an Instron-type testing machine at a nominal strain rate of 1.7 × 10⁻⁴ s⁻¹ in the temperature range between room temperature and 1000°C after holding 1 h at the test temperature in order to approach thermal equilibrium under a vacuum. Strain rate sensitivity (SRS) of flow stress was examined by the abrupt change of strain rate by a factor of ten. The effect of static strain aging on flow stress was also examined at 300°C. Slip markings were observed using an optical microscope with Nomarski interference contrast. Deformation substructures were observed by a transmission electron microscope (TEM, JEM 3010) operated at 300 kV.

3. Results and Discussion

The dominant deformation mode of [205]* oriented samples in tensile deformation was confirmed to be (001)[100] slip in the entire test temperature range. Temperature dependence of CRSS for the (001)[100] slip is shown in Fig. 1 together with the results obtained in compression using [302]* oriented samples. The CRSS showed high value at low temperatures and samples abruptly fractured before yielding in tensile test at and below 100°C. Therefore, the CRSS at low temperatures was examined in compression. Significant yielding and plastic flow were obtained above 200°C even in tension. The CRSS-temperature curve can be classified into three regimes. The CRSS rapidly decreases with increasing temperature and shows strong negative temperature dependence between 100°C and 300°C. Above 500°C, however, the CRSS anomalously increases with increasing temperature and reaches a peak around 800°C. Then, the CRSS again decreases above the peak temperature. In the temperature range where anomalous strengthening behavior occurred, sig-
significant serrations were observed on the stress-strain curves. As the anomalous strengthening behavior was advanced, the stress amplitude of the serrations increased. Above the peak temperature, serrations on stress-strain curve diminished.

Figure 2 shows the slip traces on the specimen deformed to 3% plastic strain at various temperatures with [205]* orientation in tension. Slip traces parallel to (001) are observed in all test temperatures and no twins are seen in gage section. At 300°C, fine and straight slip traces with weak contrast are homogeneously distributed. At intermediate temperatures between 500°C and 800°C, distribution of slip traces becomes inhomogeneous and the localized coarse slip bands with strong contrast are observed. At 500°C, the slip bands are initiated from specimen shoulder and propagate like Lüders band, while coarse slip bands are observed but they are nucleated homogeneously in the entire specimen surface at 800°C. At and above 900°C, the fine slip traces with weak contrast are again homogeneously distributed.

Figure 3 shows typical dislocation structures observed in specimens deformed to 3% plastic strain at various temperatures. Foils were cut parallel to the primary (001) slip plane. According to the conventional g-b contrast analysis, all dislocations have [100] Burgers vector at any test temperature. At 300°C, a large number of straight dislocations are aligned parallel to [100] and they are [100] screw dislocations. The screw dislocation can be dissociated into two 1/2[100] superpartials combined by an anti-phase boundary (APB). Since two superpartial dislocations change their contrast but the separation distance between them shows no change with different g vec-

Fig. 1 Temperature dependence of CRSS for (001)[100] slip. Tests at [302]* (▲) and [205]* (△) orientations were carried out in compression and in tension, respectively.

Fig. 2 Slip traces on the (32 0 1) surface of the specimens deformed to 3% plastic strain with [205]* orientation in tension at various temperatures (a) 300°C, (b) 500°C, (c) 800°C and (d) 1000°C.
Fig. 3  Dislocation structures in Ni$_3$Nb single crystals with [205]* orientation to 3% strain at various temperatures. (a) 300°C, (b) 700°C and (c) 1000°C. Foils were cut parallel to (001) macroscopic slip plane deformed, beam//(001). \( g = 400 \). Many waved edge segments are observed at high temperatures as indicated with small arrows.

Fig. 4  Weak beam image of dissociated [100] dislocation in Ni$_3$Nb single crystal deformed with [205]* at 300°C in tension. (a) \( g = 400, g/\text{2g} \); (b) \( g = 400, g/\text{2g} \). Foil//(001), Beam//(001).

Fig. 5  Weak beam image of [100] dislocations and loops in Ni$_3$Nb single crystal deformed with [205]* at 700°C in tension. \( g = 400, g/\text{3g} \). Foil//(001), Beam//(001).

Many small cusps are observed as indicated with small arrows in Figs. 3 and 5. Similar morphology is observed at 1000°C and the tendency to form cusps becomes remarkable.

Figure 6 shows the strain-rate dependence of flow stress at various temperatures. Tests were started at a nominal strain rate of \( 1.7 \times 10^{-4} \) s$^{-1}$ and the strain rate was abruptly changed by a factor of ten at the positions indicated with arrows in the figure. The SRS of flow stress was measured from the change in flow stress by down-changing of strain rate from \( 1.7 \times 10^{-4} \) to \( 1.7 \times 10^{-5} \) s$^{-1}$ at 1% plastic strain and was evaluated using the SRS parameter \( S = 1/T (\Delta \tau / \Delta \ln \dot{\varepsilon}) \), where \( T \), \( \tau \) and \( \dot{\varepsilon} \) indicate the test temperature, the resolved shear stress and the strain rate, respectively. Change in \( S \) with temperature is plotted in Fig. 7. The parameter \( S \) changes depending on temperature (\( T \)) and the \( S-T \) curve can be divided into three regions corresponding to the CRSS-$T$ curve.

At low temperature of 250°C, flow stress shows positive...
In the temperature range between 300°C and 700°C, SRS shows negative or little dependence. The negative dependence of SRS is known to be one of the most characteristic behavior of P-L effect. An abrupt decrease in strain rate induces the decrease in velocity of moving dislocation. If the motion of dislocation is interrupted by the dragging atmosphere of solute or impurity atoms around moving dislocation, a drop of dislocation velocity causes the easy formation of the dragging atmosphere resulting in increase of the flow stress. Strong negative dependence of SRS was obtained at 300°C although the stress-strain curve showed no frequent serrations and anomalous strengthening behavior was not significant at this temperature.

Static strain aging test was conducted at 300°C to examine the effect of the dragging atmosphere on the motion of dislocation. Firstly, specimen was deformed to 1% plastic strain, and the applied stress was unloaded to 100 MPa. After holding various periods at 100 MPa in order to examine the static aging effect, stress was again applied at 300°C. Some difference in stress before and after static aging was observed as shown in Fig. 8. The longer the aging time, the more the stress increases. This suggests that the formation of dragging atmosphere around dislocation has already occurred even at 300°C although the formation speed is too slow to induce serration (i.e. dynamic strain aging). Hence, negative SRS was observed when the strain rate was down.

At high-temperatures above 900°C, large yield drop called high-temperature yielding phenomenon was observed. This is due to the sudden multiplication of a large number of dislocations in the condition of viscous motion of dislocations with the dragging atmosphere. The serration on the stress-strain curve disappeared and SRS exhibited strong positive dependence because diffusion of atoms forming the dragging atmosphere is so fast to move together with the mobile dislocations.

As shown in Figs. 3 and 5, the dislocation lines near edge segments were heavily waved and many slight cusps were observed at high temperatures. These cusps may arise from the irregular motion of dislocation due to the dragging atmosphere and/or high frequency of climb process. Similar mor-
phology of dislocation with slight cusps was observed in FeAl in which vacancies displayed a role for dynamic aging.11)

It was revealed in this study that (001)[100] slip displayed anomalously large increase in CRSS as well as (010)[100] slip.3,5) Both slip systems have the same Burgers vector of [100], but the strengthening mechanisms are different depending on the slip planes. The anomalous strengthening for (001)[100] and (010)[100] is basically derived from the dynamic strain aging (P-L effect) and the K-W locking of screw dislocation, respectively. This indicates that the core structure of the [100] dislocation strongly controls the deformation behavior in Ni3Nb crystals. At low temperatures, CRSS for the slip on (001) exhibits relatively high value accompanied by its strong negative temperature dependence, while small value is obtained for the slip on (010).3) This suggests that the dislocation cores of [100] dislocations on (001) and (010) intrinsically have non-planar and planar structures, respectively. The difference in core structures of [100] dislocations on both planes may result in different anomalous behavior.

Although the strengthening mechanisms are different, the anomalous strengthening peaks for both slip systems appeared around 800°C. The K-W locking of [100] screw dislocation is believed to be caused by microscopic cross-slipping from (010) onto (001) by the anisotropy of APB energy.5) Hence, the mobility of [100] dislocation on (001) might affect on the unlocking behavior of K-W locked segments. More study on correlation between (010)[100] and (001)[100] slip behavior is now in progress.

4. Conclusions

Plastic behavior deforming by the (001)[100] slip in Ni3Nb single crystal with the D02 structure was investigated by tensile test at [205]* orientation. The CRSS, strain rate sensitivity of flow stress, stress-strain curve, slip line morphology and dislocation structure depended strongly on the test temperature and variations of their behavior were classified into three regions. At low temperature, motion of [100] dislocation on (001) plane was controlled by Peierls mechanism. At the intermediate temperature between 500°C and 800°C, anomalous increase in CRSS was observed. This is caused by dynamic strain aging, so-called P-L effect. Dislocations show viscous motion with dragging atmosphere at high temperature and CRSS rapidly decreased with increasing temperature.

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