Pseudoelastic Behavior of Fe–Al Polycrystals

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Pseudoelastic behavior of Fe–Al polycrystals at room temperature was examined focusing on Al concentration, the crystallographic texture and the grain size. Perfect pseudoelasticity derived from the reversible motion of 1/4(111) superpartial dislocations dragging the anti-phase boundaries (APB) took place in Fe–25.0 at% Al polycrystals at a total strain of 1.0%. The amount of strain recovery in Fe–Al polycrystals showed a maximum at 25.0 at% Al and the deviation from the concentration led to a decrease in strain recovery. The backward stress due to APB and dislocation configuration in Fe–Al alloys was closely related to the dependence of the pseudoelasticity on Al concentration. The pseudoelastic behavior of Fe–25.0 at% Al polycrystals depended strongly on the loading axis, which could be accounted for in terms of the Taylor factor. Moreover, recovery strain of Fe–23.0 at% Al polycrystals increased with increasing average grain size suggesting that the grain boundaries suppressed the forward and backward motion of the superpartials and were harmful for the pseudoelasticity.

KEY WORDS: Fe$_3$Al; intermetallic compounds; dislocations; crystallographic texture; grain size.

1. Introduction

Shape memory alloys are known to exhibit shape memory effect and pseudoelasticity by which an applied strain recovers during heating and unloading, respectively.1) In general, pseudoelasticity originates from a stress-induced martensitic transformation during loading and the reverse transformation during unloading, respectively. However, Fe–23.0Al (at%) single crystals with the D0$_3$ structure (Fe$_3$Al lattice) demonstrated perfect pseudoelasticity at room temperature regardless of the martensitic transformation.2–4) It is interesting to note that the pseudoelasticity of Fe–Al alloys was based on the peculiar dislocation motion in the D0$_3$ lattice. The (111) superdislocation was generally dissociated into four 1/4(111) superpartial dislocations bound by the nearest-neighbor and the next-nearest-neighbor antiphase boundaries (NNAPB and NNNAPB). In Fe–23.0Al single crystals, the leading 1/4(111) superpartials moved individually dragging the NNAPB during loading. During unloading, the NNAPB pulled back the superpartials due to its tension resulting in the pseudoelasticity. Therefore, we call this phenomenon “APB pseudoelasticity”.

In general, mechanical properties of polycrystalline materials depend strongly on the microstructure such as grain size and crystallographic texture.5) For instance, a yield stress of polycrystals increases with decreasing grain size through the Hall–Petch law since grain boundaries usually suppress the dislocation motion. Therefore, mechanical properties of polycrystals are generally different from those of the single crystals. Although the pseudoelastic behavior of Fe$_3$Al single crystals was extensively investigated, no systematic study using the polycrystals has not been carried out yet. In this paper, we report the pseudoelastic behavior of Fe–Al polycrystals focusing on Al concentration, the crystallographic texture and the grain size.

2. Experimental Procedure

Master ingots of Fe–16.0–28.0Al (at%) were prepared by melting high purity Fe and Al in a plasma arc furnace in a purified Ar atmosphere. The ingots 12 mm in thickness were hot-rolled to 70% reduction at 900°C and then warm-rolled to 80% at 600°C. After the rolling, the specimens were annealed at 700–1000°C for 1 h for recrystallization followed by slow cooling to room temperature at a cooling rate of 100°C/h for the D0$_3$ ordering. The microstructure of Fe–Al alloys was well documented in our previous paper.6) At and above 23 at% Al, the D0$_3$ single phase developed and there existed numerous ordered domains. In contrast, the (bcc+D0$_3$) two-phase microstructure or short-range order was observed below 23 at% Al. The tensile specimens of which gauge dimension was 1.5×0.7×5.0 mm$^3$, were cut from the recrystallized sheets by spark machining. The angle between loading axis and the rolling direction (θ) was set to be 0°, 45° and 90°. Cyclic tensile tests with incremental strain amplitudes (ε$_e$) were performed in air at room temperature at constant cross-head speed of 0.05 mm/min during loading and unloading. The applied strain amplitude was measured by a charge-coupled devises and was increased 1.0% per cycle. Note that the cross-head speed corresponded to an initial strain rate of 1.7×10$^{-4}$/s. The amount of strain recovery was evaluated using recovery ratio (R) defined as follows:
where $\varepsilon_t$ and $\varepsilon_r$ are the total strain and the residual strain, respectively. Microstructure of the specimens was analyzed by an electron back-scatter diffraction (EBSD) method. The electron beam was automatically moved in $10 \, \mu m$ steps to develop an orientation map (OIM).

3. Results

3.1. Microstructure of Fe–Al Polycrystals

Figure 1 shows OIM maps of Fe–23.0Al warm-rolled sheets before and after recrystallization at 800°C for 1 h. In as warm-rolled specimens, there exists two types of deformation bands oriented (111) and (110) directions parallel to the normal and rolling directions (ND and RD), respectively (Figs. 1 (a), 1(b)). This means the $\{110\}$ and $\{111\}$ fiber textures develop in the as warm-rolled Fe–23.0Al sheets. After the recrystallization treatment at 800°C, the frequency of grains belonging to the $\{111\}$ fiber texture increases as shown in Figs. 1 (c) and 1(d). It is also noted that the equiaxed grains of which the average grain size ($\bar{d}$) is 43.6 $\mu m$ can be observed after annealing at 800°C. Such rolling and recrystallization textures are similar to those of bcc metals.

Figure 2 shows orientation distribution function (ODF) at $\phi_2=45°$ for Fe–16.0–28.0Al polycrystals after recrystallization at 800°C for 1 h. Below 23 at% Al, development of near-Cube texture ($\phi_1=45°$, $\phi=0°$) can be seen as shown in Figs. 2 (a) and 2(b). In contrast, the intensity around $\phi=55°$ becomes strong at and above 23 at% Al suggesting that the $\{114\}$ component which generally develops in bcc metals at higher reduction in thickness.

The $\bar{d}$ of Fe–23.0Al polycrystals recrystallized at 700–1000°C was measured using the EBSD technique. The $\bar{d}$ increased from 26.9 to 140.6 $\mu m$ with increasing recrystallization temperature from 700 to 1000°C.

3.2. Pseudoelastic Behavior of Fe–Al Polycrystals

Stress–strain (S–S) curves of Fe–25.0Al polycrystals recrystallized at 800°C for 1 h and then pulled with $\theta=0°$ at room temperature are shown in Fig. 3. In the repeated tensile tests, $\varepsilon_t$ was increased 1.0% per cycle. At $\varepsilon_t=1.0\%$, perfect pseudoelasticity takes place in the Fe–25.0Al polycrystals. On the other hand, the residual strain becomes large as $\varepsilon_t$ increases and the S–S curve shifts to higher strains. Note that the recoverable strain of Fe–23.0Al single crystals was approximately 5.0%, indicating that the pseudoelastic properties of Fe–Al polycrystals were inferior to those of the single crystals. However, in other words, a recoverable strain of 1.0% could be obtained even in the Fe–Al polycrystals. The $R$ values of Fe–Al alloys at $\theta=0°$ and 90° after recrystallization at 800°C for 1 h are plotted against Al concentration in Fig. 4. The $R$ values at $\varepsilon_t=1.0$ and 2.0% show a maximum at 25.0 at% Al both at $\theta=0°$ and 90° (Figs. 4(a) and 4(b)) and decreases with increasing the deviation from the stoichiometric concentration. In the

$$R = \frac{\varepsilon_t - \varepsilon_r}{\varepsilon_t} \times 100 \quad \text{................(1)}$$

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case of Fe–Al single crystals, however, the optimum Al concentration of the pseudoelasticity was 23 at%, different from that in the polycrystals.4)

The $R$ value of Fe–25.0Al polycrystals recrystallized at 800°C for 1 h and then tensile deformed to $\varepsilon_{t}=1.0$, 2.0 and 3.0% at room temperature. $R$ value at $\theta=0^\circ$ is closely related to $\langle 0001 \rangle$ preferred orientation.11}

Figure 6 shows relationship between $\bar{d}$ and $R$ value in Fe–23.0Al polycrystals tensile deformed to $\varepsilon_{t}=3.0\%$ at room temperature. Fe–23.0Al polycrystals annealed at 700°C with $\bar{d}=26.9\ \mu m$ showed small $R$ value. However, the $R$ value increases with increasing $\bar{d}$ and reaches to 45% with $\bar{d}=140.6\ \mu m$. Thus, the pseudoelasticity of Fe–Al crystals is closely related to $\bar{d}$.

4. Discussion

In the present study, we first found that not only Fe–Al single crystals but also the polycrystals demonstrated large pseudoelasticity. In particular, $\varepsilon_{t}=1.0\%$ was recoverable in Fe–25.0Al polycrystals. At present, there was no indication that martensites or twins were responsible for the pseudoelasticity in Fe–Al polycrystals. Thus, it is reasonable to consider that APB pseudoelasticity appeared in the poly-

Figure 4. Dependence of $R$ on Al concentration in Fe–Al polycrystals recrystallized at 800°C for 1 h and then tensile deformed to $\varepsilon_{t}=1.0$ and 2.0% with $\theta=0^\circ$ (a) and 90° (b) at room temperature.

Figure 5. Changes in $R$ and $\sigma_{y}$ with $\theta$ in Fe–25.0Al polycrystals recrystallized at 800°C for 1 h and then tensile deformed to $\varepsilon_{t}=1.0$, 2.0 and 3.0% at room temperature.

Figure 6. Variation in $R$ with $\bar{d}$ in Fe–23.0Al polycrystals pulled to $\varepsilon_{t}=3.0\%$ at room temperature; $\theta=0^\circ$.
large pseudoelasticity took place at \( x = 0.23 \) and 0.25. In contrast, the \( R \) above \( x = 0.25 \) exhibited small values in contrast to high \( \tau_{\text{NNN}} \). This is because the dislocation configuration changed above \( x = 0.25 \); paired \( 1/4 \langle 111 \rangle \) superpartials started to move dragging the NNAPB.\(^{3,4} \) The backward stress of the NNAPB (\( \tau_{\text{NNN}} \)) is given by\(^{7} \)

\[
\tau_{\text{NNN}} = \frac{\gamma_{\text{NNN}}}{2b}
\]

where \( \gamma_{\text{NNN}} \) is the NNAPB energy written as:

\[
\gamma_{\text{NNN}} = \frac{2\sqrt{2}}{d_0^2} \{2V_2S_2^2\}
\]

The \( \tau_{\text{NNN}} \) is also shown in Fig. 7. Note that the denominator of Eq. (6) is two times larger than that of Eq. (2) while the \( \gamma_{\text{NNN}} \) shows similar value to the \( \gamma_{\text{NN}} \) at around \( x = 0.25 \). As a result, the \( \tau_{\text{NNN}} \) is much smaller than the \( \tau_{\text{NN}} \) resulting in small \( R \) above \( x = 0.25 \). Therefore, the dependence of pseudelastic behavior in Fe–Al polycrystals on Al concentration could be interpreted in terms of the backward stress originating from the NNAPB and the dislocation configuration.

It should be noted that Fe–Al single crystals demonstrated maximum \( R \) at 23.0 at% Al while the optimum Al concentration for the pseudoelasticity in the polycrystals was 25.0 at% Al.\(^{3,4} \) In Fe–Al single crystals, the dislocation configuration of \( 1/4 \langle 111 \rangle \) superpartials strongly affected the pseudelastic behavior. The individual motion of \( 1/4 \langle 111 \rangle \) superpartials dragging the NNAPB resulted from the interaction between the superpartials and the boundaries of the \( D_0 \) ordered domains.\(^{3,4} \) The fine ordered domains less than 50 nm in diameter, developed at 23.0 at% Al were optimum for \( 1/4 \langle 111 \rangle \) superpartials moving independently, resulting in the maximum \( R \) at the Al content in the single crystals. On the other hand, the domain size increased with increasing Al content and consequently, the activation of paired \( 1/4 \langle 111 \rangle \) superpartials dragging the NNAPB led to the decrease in \( R \).\(^{3,4} \) In Fe–Al polycrystals, however, the grain boundaries suppressed the motion of \( 1/4 \langle 111 \rangle \) superpartials. Furthermore, several slip systems were necessarily activated in the polycrystals to transfer strain across the grain boundaries. The interaction between the activated slip systems resulted in the formation of sessile dislocations. Consequently, the frictional stress of \( 1/4 \langle 111 \rangle \) superpartials in Fe–Al polycrystals became larger than that of the single crystals. Under such circumstances, higher \( \tau_{\text{NNN}} \) was required for the backward motion of the superpartials in Fe–Al polycrystals, compared with the single crystals. The \( \tau_{\text{NNN}} \) at 25.0 at% Al was higher than that at 23.0 at% Al by 30 MPa (Fig. 7), which may result in the highest \( R \) in Fe–25.0Al polycrystals.

The strain recovery of Fe–Al polycrystals was much lower than that of the single crystals (Figs. 3 and 4). For instance, the recoverable strain of Fe–23.0Al polycrystals was below 1.0%, much smaller than that of the single crystals. It is noted that Fe–23.0Al single crystals oriented for single slip exhibited a maximum recoverable strain.\(^{8} \) In contrast, in a polycrystalline material, a sufficient number of independent slip systems are required to accommodate strain at a grain boundary. The activated slips in Fe–Al polycrystals interacted with each other resulting in the formation of sessile dislocations. Moreover, the grain boundaries suppressed the reversible motion of \( 1/4 \langle 111 \rangle \) superpartials dragging the NNAPB. Thus, the formation of the sessile dislocations and the grain boundary effect led to the decrease in \( R \) in the polycrystals. In addition, in Fe–23.0Al polycrystals, the \( R \) rose with an increase in \( d \) as shown in Fig. 6. As the \( d \) of the polycrystals increased, the effect of the grain boundaries became small, which was suitable for the pseudelasticity. However, the elongation of Fe–Al polycrystals decreased with increasing \( d \) due to the stress concentration at the grain boundaries. Therefore, an optimization of \( d \) is necessary for obtaining good balance between the pseudelasticity and elongation.

The effect of crystallographic texture on the pseudoelasticity was discussed. In Fe–Al polycrystals, the recrystallization texture at 800°C varied from near-Cube to the \( \gamma \) fiber textures with increasing Al concentration as shown in Fig. 2. Below 23 at% Al where the near-Cube texture developed, the \( \tau_{\text{NN}} \) was so small that reversible motion of \( 1/4 \langle 111 \rangle \) superpartials hardly occurred irrespective of the texture (Fig. 7). Therefore, the texture transition was insignificant for the pseudelastic properties of Fe–Al polycrystals. On the other hand, the pseudelasticity of Fe–25.0Al polycrystals showed strong \( \theta \) dependence (Fig. 5). The \( R \) and \( \sigma_\gamma \) at \( \theta = 0°, 90° \) was much higher and lower than that at \( \theta = 45° \), respectively. The dependence of \( R \) and \( \sigma_\gamma \) on \( \theta \) was found to correlate with Taylor factor (\( M \)) which could be obtained by the OIM software. Fe–25.0Al polycrystals warm-rolled and annealed at 800°C exhibited the \( \gamma \) fiber texture where \( \langle 111 \rangle \) direction of the grains was oriented parallel to the ND. Although the formation of the \( \gamma \) fiber texture generally led to isotropic deformation in the polycrystalline sheets compared with the other texture components, plastic anisotropy depending on \( \theta \) was reported in some bcc metals.\(^{9} \) For instance, both the \( \sigma_\gamma \) and \( M \) of the Al-killed steels having the \( \gamma \) fiber texture showed a maximum at \( \theta = 45° \). In the present study, the \( M \) of Fe–25.0Al polycrystals was also largest at \( \theta = 45° \) among the three loading axes similar to the bcc metals (Fig. 8). Moreover, it is natural that large \( M \) at \( \theta = 45° \) resulted in higher \( \sigma_\gamma \). The orientation dependence of the pseudelasticity of the Fe–Al single crystals was systematically examined in our previous work.\(^{10} \) In the single crystals, the \( R \) increased with increasing Schmid factor for primary
\{101\}\{111\} slip. If the Schmid factor decreased, 1/4\{111\} superpartials tended to cross slip onto \{211\} plane. Numerous dislocation dipoles which was immobile and suppressed the reversible motion of 1/4\{111\} superpartials were formed as a result of the cross slip. Thus, high Schmid factor for \{101\}\{111\} primary slips was favorable for large strain recovery in Fe–Al single crystals. The large M at \theta=45^\circ means the existence of \{101\}\{111\} slip of which Schmid factor is small, which resulted in small R at the orientation. However, the variation in R with \theta became unnoticeable after work hardening at and above \varepsilon_r=1.0\%. It should also be noted that Young’s modulus of bcc metals with the \gamma fiber texture showed a maximum at \theta=45^\circ.10) High Young’s modulus at \theta=45^\circ led to an increase in plastic strain at constant \varepsilon_r, also resulting in the decrease in R. One of the possible methods to avoid the effects of grain boundary and crystallographic texture is to control an abnormal grain growth. It is widely known that strong \{110\}\{001\} Goss texture of which orientation dispersion is within 3° can be obtained in Fe–3 mass\%Si steels with the bcc structure by controlling the grain growth.11) If such strong texture is developed in Fe–Al polycrystals, the polycrystals can be regarded as the single crystal. This may lead to the improvement of the pseudoelasticity. Since Fe–Al alloys have the D0\_3 structure based on the bcc structure, there is a possibility that the texture can be controlled by the microstructure control. Further work along this line is presently being undertaken.

5. Conclusions

The pseudoelastic behavior of Fe–Al polycrystals was examined focusing on Al concentration, the plastic anisotropy and the grain size. The following conclusions were reached.

(1) D0\_3-ordered Fe–Al polycrystals as well as the single crystals showed APB pseudoelasticity at room temperature. In particular, Fe–25.0Al polycrystals exhibited perfect strain recovery at \varepsilon_r=1.0\%. On the other hand, the R became small as the deviation from 25.0 at\% Al increased.

(2) The pseudoelasticity of Fe–25.0Al polycrystals was dependent on \theta. The R at \theta=45^\circ was lower than that at \theta=0^\circ and 90^\circ.

(3) \(R\) increased with an increase in \(\varepsilon_r\), presumably due to the suppression of the reversible motion of 1/4\{111\} superpartials by the grain boundaries.

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