In Situ Observation of Pseudoelasticity in Fe₃Al Single Crystals with D₀₃ Structure

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In order to clarify the mechanism of pseudoelasticity in Fe–23.0Al (at%) single crystals with the D₀₃ structure, in situ observation of tensile deformation was performed at room temperature using an optical microscope (OM), an electron back-scatter diffraction (EBSD) device and an X-ray Laue back-reflection (XLB) machine. In the present study, neither martensite nor twin was observed during the pseudoelasticity. In particular, the whole of the tensile specimen could be indexed with respect to the D₀₃ structure by (h111) slip planes. The area fraction of the slip bands increased linearly with increasing plastic strain up to 0.066. During unloading, the slip bands disappeared, resulting in pseudoelasticity. From in situ XLB tests, the crystal rotation of the loading axis toward [111] slip direction took place during loading. The rotation angle and the plastic strain satisfied the Schmid-Boas relationship at small plastic strains, especially for (112) and (211) slips. These results suggest that the reversible motion of 1/4(111) superpartial dislocation dragging an antiphase boundary (APB) was responsible for the pseudoelasticity in Fe–23.0Al single crystals.

Keywords: iron aluminides, pseudoelasticity, shape memory alloys, dislocation dynamics

1. Introduction

It is well known that pseudoelasticity is generally due to martensitic transformation or deformation twinning.¹ For example, in Ti–Ni alloys, stress-induced martensites disappeared during unloading at a particular temperature, resulting in perfect pseudoelasticity (superelasticity). On the other hand, Fe–Be alloys demonstrated pseudoelasticity based on twinning and untwining during loading and unloading, respectively.²,³ However, Fe–23.0Al (at%) single crystals with the D₀₃ structure exhibited a large pseudoelasticity in the temperature range between −50 and 200°C although there was no evidence that stress-induced transformation and twinning occurred.⁴–¹⁴ The pseudoelasticity was considered to arise from the peculiar dissociation behavior of (111) superlattice dislocation. The (111) superdislocation was generally dissociated into four 1/4(111) superpartials located by the nearest-neighbor and the next-nearest-neighbor antiphase boundaries (NNAPB and NNNAPB, respectively).¹⁵,¹⁶ However, when pseudoelasticity took place in Fe–23.0Al single crystals, the leading 1/4(111) superpartials moved independently dragging the NNAPB during loading.⁵–¹⁴ During unloading, the NNAPB pulled back the superpartials because of its tension resulting in the pseudoelasticity. Kubin et al.¹⁵ confirmed the reversible motion of 1/4(111) superpartials dragging the NNAPB by in situ transmission electron microscope (TEM) observation. Therefore, we term this phenomenon “APB pseudoelasticity”.

Very recently, the discussion on the mechanism of pseudoelasticity in Fe–Al alloys was revisited. Kabra et al.¹⁷ performed in situ neutron diffraction studies on deformed Fe–23.0Al single crystals and found that the peak intensity of the neutron diffraction changed during plastic deformation. They implied that twinning or phase transformation was responsible for the pseudoelasticity. In one of our previous reports, we showed that Fe–23.8Ga single crystals with the D₀₃ structure demonstrated multimode pseudoelasticity resulting from martensitic transformation, twinning and dislocation motion, depending on the degree of long-range order and loading axis.¹⁸ Thus, further investigation is necessary to clarify the mechanism of the pseudoelasticity in D₀₃-ordered Fe–23.0Al single crystals.

Because pseudoelasticity is a dynamic phenomenon under load, a post mortem study after deformation provides limited information. Therefore, in the present study, in situ observation using an optical microscope (OM), electron back-scatter diffraction (EBSD) device and X-ray Laue back-reflection (XLB) instrument was systematically carried out to understand the pseudoelastic behavior in Fe–23.0Al single crystals.

2. Experimental Procedure

Master ingots of Fe–23.0Al were prepared by melting high purity Fe and Al in a plasma arc furnace under a purified Ar atmosphere. The single crystals were grown from the ingots by a floating zone method at a growth rate of 5 mm/h. After homogenization at 1373 K for 48 h, the crystals were slowly cooled to room temperature at 80 K/h for D₀₃ ordering. The dog-bone-shaped tensile specimens for in situ OM and XLBR tests, with gauge dimensions of 1.5 × 0.8 × 5.0 mm³, were cut from the crystals by electric discharge

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machining. The loading axes ($L_A$, $L_B$ and $L_C$) and plate normals ($N_A$, $N_B$ and $N_C$) of the tensile specimens are plotted in a standard stereographic projection shown in Fig. 1. At A, B and C orientations, the primary slip systems are ($\{\overline{1}1\overline{2}\}$ [001]), ($\{1\overline{1}1\}$ [001]) and ($\{2\overline{1}\}$ [001]) with Schmid factor of 0.5, respectively. In the case of in situ OM tests, the tensile test machine was clamped on the stage of the OM equipped with a video camera and the slip markings on the sample surface were recorded in air at room temperature. Furthermore, the area fraction of the slip markings was evaluated using an image processing software. Note that the cross-head speed during loading and unloading was set to be 0.05 mm/min corresponding to an initial strain rate of $1.7 \times 10^{-4}$/s. In the case of in situ XLBR tests, the tensile test machine was placed on the sample stage of the Laue machine. The tensile specimens were irradiated with a white X-ray from a tungsten target to obtain the XLBR patterns. The initial directions of incident X-ray were parallel to $N_A$, $N_B$ and $N_C$ at A, B and C orientations, respectively (Fig. 1), although the direction varied during deformation due to crystal rotation. Since it took 40 s to take an XLBR photograph, the cross-head of the tensile test machine was temporarily stopped during the X-ray irradiation.

Tensile tests at B orientation were also done in a scanning electron microscope (SEM, JSM7000F, JOEL) equipped with an EBSD device (EDAX, Inc.). An initial strain rate for the tensile test was set to be $3.2 \times 10^{-3}$/s. During the EBSD measurement, a cross-head of the tensile test machine (Ernest F. Fullam, Inc.) was stopped and an electron beam was automatically moved on the $N_B$ plane in 1.5 µm steps to develop an orientation map.

3. Results

3.1 Stress–strain response

Figure 2 shows the stress–strain curves of Fe–23.0Al single crystals pulled to a plastic strain ($\varepsilon_p$) of 0.050 with A, B and C orientations at room temperature. Fe–23.0Al single crystals demonstrate incomplete pseudoelasticity at A orientation; only half of the applied strain is recovered during unloading. In contrast, perfect pseudoelasticity can be obtained at B orientation. The stress–strain curve at B orientation is characterized by a steady-state flow without a serration during loading and a huge strain recovery during unloading. On the other hand, a little strain remains after unloading at C orientation. It should be mentioned that the yield stresses at A and C orientations are lowest and highest among the orientations, respectively.

The stress–strain curve of Fe–23.0Al single crystals tensile deformed at B orientation to fracture is shown in Fig. 3. After yielding, the stress level increases gradually with increasing strain followed by a small decrease near the inflection point ($\varepsilon_p \approx 0.15$) indicated by arrows. Further increase in $\varepsilon_p$ results in a small stress increase and finally, the flow stress gradually decreases due to a crack initiation.

3.2 In situ observation using OM

Figure 4 shows the slip traces of the specimens tensile deformed to $\varepsilon_p = 0.050$ at A, B and C orientations. Before deformation, a mirror surface finished by electropolishing can be seen at all orientations (Figs. 4(a), 4(e), 4(i)). The formation of slip traces starts at the onset of plastic deformation, especially near the border between the round and gauge sections in the tensile specimens (Figs. 4(b), 4(f), 4(j)). At any orientation tested, slip traces perpendicular to the loading axis can be seen. The slip planes were determined...
by two-surface analysis to be primary (1\bar{1}2), (1\bar{0}1) and (2\bar{1}1) slips at A, B and C orientations, respectively. In particular, thick slip bands showing a black contrast are nucleated at B orientation, as shown in Fig. 4(f). As deformation proceeds, the slip bands at B orientation thicken and new bands are nucleated. In contrast, the slip traces and slip bands lose their contrast gradually during unloading (Figs. 4(c), 4(g), 4(k)) and almost disappear at zero stress (Figs. 4(d), 4(h), 4(l)), though some traces remain in the other area at A and C orientations.

Since the slip bands at B orientation were clearly visible, the area fraction of the bands ($f$) was quantitatively evaluated by an image processing software. As shown in Fig. 5, $f$ is obviously proportional to $\varepsilon_p$, and the following equation holds true:

$$\varepsilon_p = f\varepsilon_{\text{band}}$$

(1)

where $\varepsilon_{\text{band}}$ is the individual strain of the slip bands. $\varepsilon_{\text{band}}$ can be calculated to be 0.066 by an extrapolation to $f = 1$. In other words, the specimen surface can be fully covered by the slip bands at $\varepsilon_p = 0.066$.

Figure 6 shows an OM micrograph of Fe–23.0Al single crystals pulled to $\varepsilon_p = 0.150$; at room temperature, B orientation.

3.3 In situ observation using SEM-EBSD

Figure 7 shows the inverse pole figure (IPF) maps of Fe–23.0Al single crystal tensile deformed at B orientation, which was obtained by in situ SEM-EBSD observation. The color of the maps corresponds to the tensile axis. During loading, the map color changes gradually from peach to yellow due to a crystal rotation (Figs. 7(a)–7(d)). On the other hand, the reverse crystal rotation occurs during unloading, as shown in Figs. 7(e)–7(h). In addition, the whole of the tensile specimen can be indexed with respect to the D03 structure suggesting that neither martensitic transformation nor twinning takes place. The tensile axis during loading and unloading is
plotted against a unit triangle in Fig. 8. During loading, the tensile axis rotates toward [111] direction along [1/C2210] zone while the reverse rotation occurs during unloading. Consequently, the tensile axis returns to the initial position after complete unloading. In Fig. 9, the crystal rotation from the initial orientation is visualized by a color gradient (Fig. 9(i)). During loading (Figs. 9(a)–9(d)), a banded structure corresponding to the slip bands shown in Fig. 4(f) appears in the crystal and propagates from right to left with increasing $\varepsilon_p$. Large crystal rotation preferentially occurs in the slip bands. At $\varepsilon_p = 0.043$, the rotation angle reaches about $3^\circ$ (Fig. 9(d)). In contrast, the banded structure disappears and the crystal returns to the initial orientation during unloading, as shown in Figs. 9(e)–9(h).

### 3.4 In situ observation using XLBR machine

In situ observation using an XLBR instrument was performed to examine the crystal rotation during loading to higher strains. The variation in tensile axis during loading, examined from the XLBR patterns, is shown in a stereographic projection in Fig. 10. During tensile loading, the loading axes rotate toward [111] slip direction along [1/C2210], [1/C2221] and [0/C2211] zones at A, B and C orientations, respectively (Figs. 10(b), 10(c), 10(d)). This is consistent with the crystal rotation by [111] slip deformation. Moreover, the rotation angle toward [111] slip direction ($\Delta \lambda$) is plotted against $\varepsilon_p$ as shown in Fig. 11. When slip deformation occurs in a crystal, $\Delta \lambda$ and $\varepsilon_p$ are known to satisfy the Schmid-Boas (S-B) relationship given below:\(^{19}\)

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Fig. 7 IPF maps for tensile axis of Fe–23.0Al single crystal pulled with B orientation at room temperature; *in situ* SEM-EBSD observation. (a)–(d) During loading, (e)–(h) during unloading. (a) Before loading (the surface normal is nearly $N_B$ shown in Fig. 1), (b) $\varepsilon_p = 0.009$, (c) $\varepsilon_p = 0.027$, (d) $\varepsilon_p = 0.043$, (e) $\varepsilon_p = 0.039$, (f) $\varepsilon_p = 0.017$, (g) $\varepsilon_p = 0.007$, (h) after unloading. (i) A unit triangle showing color code.

Fig. 8 Variation in tensile axis during pseudoelasticity in Fe–23.0Al single crystal pulled with B orientation at room temperature; *in situ* SEM-EBSD observation. (a)–(h) correspond to those in Fig. 7.

Fig. 9 Variation in rotation angle of the tensile axis of Fe–23.0Al single crystal pulled with B orientation at room temperature; *in situ* SEM-EBSD observation. The color gradient corresponds to the rotation angle from the initial orientation, as shown in (i). (a)–(d) During loading, (e)–(h) during unloading. (a) Before loading (the surface normal is nearly $N_B$ shown in Fig. 1), (b) $\varepsilon_p = 0.009$, (c) $\varepsilon_p = 0.027$, (d) $\varepsilon_p = 0.043$, (e) $\varepsilon_p = 0.039$, (f) $\varepsilon_p = 0.017$, (g) $\varepsilon_p = 0.007$, (h) after unloading.
\[
\exp(\varepsilon_p) = \frac{\sin \lambda_0}{\sin \lambda_0 - \Delta \lambda}
\]

where \(\lambda_0\) is the initial angle between the loading axis and [111] slip direction (i.e., \(\lambda_0 \approx 45^\circ\)). \(\Delta \lambda - \varepsilon_p\) curves at A (Fig. 11(a)) and C (Fig. 11(c)) orientations are in quantitatively good agreement with the S-B relationship although the curves gradually shift toward small rotation angles probably due to the activation of the other slip systems. This strongly suggests that the crystal rotation in Fe\textsubscript{23.0}Al single crystals during loading is caused by [111] slip deformation. On the other hand, the crystal rotation at B orientation is complicated; the rotation behaviors in the two in situ XLBR tests are different from each other as shown in Fig. 11(b). In the first test, crystals rotate rapidly at \(\varepsilon_p \leq 0.02\) and then the \(\Delta \lambda - \varepsilon_p\) curve converges to the ideal S-B line. In the second test, however, little rotation is observed up to \(\varepsilon_p = 0.04\) followed by a rapid increase in \(\Delta \lambda\) by further deformation. The difference in rotation behaviors between the two tests is closely related to the strain localization at the coarse slip bands, as shown in Figs. 4(f) and 9(c).

4. Discussion

In our previous study, Fe–23.8Ga single crystals with the D\(_0\text{3}\) structure were found to demonstrate transformation and twinning pseudoplasticities as well as APB one depending on the degree of D\(_0\text{3}\) order and the loading axis.\(^{18}\) In Fe–23.8Ga alloys, the habit planes of the stress-induced martensites and the deformation twins were \(\{331\}\) and \(\{211\}\), respectively. The surface relief by the martensites and the twins was quite different from that by \(\{101\}\) and \(\{211\}\) slip traces observed in the present study. Therefore, one can conclude that the surface relief observed on Fe–23.0Al crystals was due to slip deformation by the dislocation motion. In addition, the crystal rotation toward [111] slip direction occurred during loading, obeying the Schmid-Boas relationship, especially at A and C orientations (Figs. 7–11). This strongly suggests that the motion of 1/4[111] superpartial dislocations was responsible for the deformation. In fact, 1/4[111] superpartials were observed in Fe–23.0Al single crystals at A–C orientations.\(^{10}\) In contrast, neither martensitic transformation nor twinning seems to take place in the course of deformation. For instance, a serrated flow accompanied by deformation twinning could not be observed in the stress–strain curve in Fe–23.0Al crystals, as shown in Fig. 2. In the neutron diffraction study carried out by Kabra \textit{et al.}\(^{17}\) the peak intensity of the neutron diffraction varied during loading and unloading. However, the Fe\textsubscript{3}Al specimen used in the neutron diffraction study was a single crystal. The crystal rotation by slip deformation could cause the change in the peak intensity in the single crystal. Consequently, the result of the neutron study could not prove the occurrence of martensitic transformation and twinning. Thus, it is strongly suggested that the APB pseudoplasticity based on the reversible motion of 1/4[111] superpartials dragging the NNAAPB appeared in D\(_0\text{3}\)-ordered Fe–23.0Al single crystals. It should be mentioned that D\(_0\text{3}\)-ordered Fe\textsubscript{2}Ga single crystals exhibited multimode pseudoplasticity based on slip...
The area inside the loops was proposed, as shown in Fig. 12. In Fe₃Ga crystals, the formation of slip markings means that some dislocations passed through the specimen surface. One of the possible mechanisms which can interpret the reversible slip passing through the surface was deformation, martensitic transformation and deformation twinning. In contrast, Fe₃Al single crystals exhibited only APB pseudoelasticity though the crystals also had the D₀₃ structure. The ordering energy and the degree of D₀₃ order in Fe₃Ga were higher than those of Fe₃Al, which may explain the difference in their pseudoelastic behavior.

The formation of the slip markings in Fe₃₋₋₋₀₁₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋˓...

**Fig. 12** Relationship between slip trace formation and dislocation motion in Fe₃₋₋₋₀₁₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋₋之內容一Backdrop. (a) 1/4(111) superpartial loop elongating along the screw direction expand, (b) a part of the loops passes through the surface and (c) remaining part of the loops shrink.
ever, since the APB pseudoelasticity is based on dislocation motion, the physical meaning of $\varepsilon_{\text{max}}$ is still unclear. Therefore, further study should be needed for a complete understanding.

At B orientation, the activation of the secondary (101) slip was suppressed below the inflection point (Fig. 3), suggesting that a strong latent hardening took place in Fe–23.0Al single crystals. It is also noted that such latent hardening was also observed at A and C orientations, though the results are not shown here. If the APB pseudoelasticity appeared in Fe–23.0Al crystals pulled at B orientation, 1/4[111] superpartials moved individually dragging the NNAPB on the primary (101) plane. From the crystallographic viewpoint, the secondary (101) slip must cut the NNAPB on the primary slip planes. It is natural that the superpartials on the secondary (101) plane also trail the NNAPB. Therefore, an intersection of the NNAPBs on the primary (101) and secondary (101) slips occurred. Yoshimi et al.\textsuperscript{23) reported that such intersection of the NNAPBs led to strong work hardening since Al cluster was geometrically created at the intersection. Thus, the NNAPB on the primary (101) plane acted as a strong barrier to the secondary (101) slip, which led to the latent hardening. Since the dislocation interaction between the different slips was harmful to the reversible motion of dislocations, the planar single slip associated with strong latent hardening was favorable for the pseudoelasticity. Once the secondary (101) slip passed through the primary (101) slip plane, the slip propagated catastrophically with the frequent cross-slip to (211) plane. This resulted in the stress drop at the inflection point shown in Fig. 3.

5. Conclusions

In situ observation of pseudoelasticity in D0\textsubscript{3}-ordered Fe–23.0Al single crystals was performed to clarify the mechanism of the pseudoelasticity. The following conclusions were reached.

1) Some in situ observations done in the present study suggested that the APB pseudoelasticity based on reversible motion of 1/4[111] superpartials dragging the NNAPB appeared in D0\textsubscript{3}-ordered Fe–23.0Al single crystals at room temperature. In contrast, there was no indication of stress-induced martensitic transformation or twinning in the crystals.

2) (112), (101) and (211) slips were observed to occur depending on the loading axis. All these slips contributed to the APB pseudoelasticity and the recovery strain by (101) slip was largest among the slips.

3) At B orientation at which (101) slip occurred, the slip bands with an individual strain of 0.066 were formed during deformation. The area fraction of the slip bands increased linearly with increasing plastic strain, accompanied by the band thickening and nucleation of new bands.

4) Crystal rotation toward [111] slip direction during loading was observed at any loading axis tested. The rotation angle and the plastic strain satisfied the Schmid-Boas relationship at small strains, especially at A and C orientations.

5) Strong latent hardening caused by the interaction between the NNAPBs on the primary and secondary slip planes took place in D0\textsubscript{3}-ordered Fe–23.0Al single crystals, which was suitable for the occurrence of the pseudoelasticity.

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