Tensile deformation and recrystallization of aluminum single crystals with sub-grained structures studied by synchrotron X-ray radiation

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Received: 6 December 2019; Revised: 21 January 2020; Accepted: 12 March 2020

Abstract
X-ray diffraction experiments were carried out to non-destructively study the interior of aluminum single crystals at the pre-deformation, post-deformation and post-annealing stages using synchrotron radiation at SPring-8. Single crystals were grown by the Bridgman method. The as-grown samples were not perfect from the crystallographic perspective, but were composed of several sub-grains with misorientations smaller than 1° across the sub-boundaries. Although the difference in orientation in the cross section of the sample increased about 0.7° by the application of the nominal strain of 8% in tension along the <111> direction, the sub-grain structures remained largely unchanged. No evidence was found of the local accumulation of dislocations at the sub-boundaries. The orientation map of the deformed sample clearly shows that no non-uniformly deformed regions formed inside the sample. The level of residual strain calculated by the strain-scanning method was as low as 1×10⁻⁴. After annealing at 480 °C, the whole cross section of the center of the sample was covered by a single recrystallized grain. Although the resolution of the present experiment was insufficient to observe potential nucleation sites of recrystallized grains in <111>-oriented crystals, a possible candidate for the sites may be assumed at the intersections of certain types of slip bands.

Keywords: Aluminum, Single crystal, Sub-grain, Tensile deformation, Recrystallization, Synchrotron radiation

1. Introduction

Recrystallization is the most important annealing phenomenon in plastically deformed metals, in which grains with low defect density are nucleated and grow by consuming the surrounding deformation matrix. The newly formed grains are referred to as recrystallized grains, and the difference in orientation between them and the deformation matrix is often a few tens of degrees or more. Deformation and recrystallization have long been a focus of research, since they play fundamentally important roles in the control of polycrystalline metal (e.g., control over grain size and preferred crystallographic orientation of grains).

Since both deformation and recrystallization are complex phenomena, efforts have been made to reduce the number of correlated factors in experimental studies. One approach to excluding unwanted factors is to use single-crystalline samples instead of polycrystalline ones. Single-crystalline samples make it possible to exclude the influence of complex deformation close to the grain boundaries. Some research groups have reported the development of deformation-induced microstructures and recrystallization in aluminum-based single crystals deformed by channel die compression (Ferry and Humphreys, 1996; Stanford et al., 2003; Paul, 2003; Miszczyk et al., 2017). Pure metals
without particles and precipitates are more favorable than alloys. In addition, tensile deformation is preferable to rolling, because it excludes the need to take into account the influence of complex shear deformation on the sample surface induced by friction with the rollers. Hence, recrystallization in single-crystalline samples of pure metals deformed in tension is the simplest form of experimental study (Yoshida et al., 1959; Senna and Lücke, 1976). Working from this concept, Inoko’s research group has carried out systematic studies using pure aluminum (Al) single crystals deformed in tension (Inoko et al., 1994; Kashiwara et al., 1996a, 1996b; Kashiwara et al., 2000; Okada et al., 2001; Tagami et al., 2001; Wert et al., 2003; Okada et al., 2003; Inoko et al., 2010; Inoko et al., 2011a, 2011b).

The deformation behavior of Al single crystals changes with tensile orientation. Crystals can be classified into two groups, with or without the formation of bands of non-uniformly deformed regions. For example, when a single crystal is deformed in tension along the \(<1\ 4\ 10>\) direction, only one slip system (i.e., the primary slip) is initially activated, and kink bands are introduced to accommodate the rotation of the deformation matrix (Inoko et al., 1994). Similarly, if the tensile axis is parallel to the \(<1\ 1\ 0>\) direction, special bands of secondary slip are formed (Kashiwara et al., 1996a, 1996b; Okada et al., 2001; Wert et al., 2003; Okada et al., 2003). On the other hand, non-uniformly deformed regions are unlikely to be formed when single crystals are deformed in tension along the \(<1\ 1\ 1>\), \(<1\ 1\ 2>\) or \(<0\ 0\ 1>\) direction (Kashiwara et al., 2000; Tagami et al., 2001). In single crystals of these orientations, multiple slip systems are activated from the initial stage of deformation, and the crystallographic orientation remains unchanged with strain. Hence, such tensile orientations are referred to as uniformly deformed orientations.

It is known that work hardening and recrystallization behaviors are not identical in all uniformly deformed orientations. The largest work hardening is observed in single crystals deformed in tension along the \(<1\ 1\ 1>\) direction, due to the suppression of long-range cross slip (Tagami et al., 2001). Hereafter, we refer to such single crystals as \(<1\ 1\ 1>\)-oriented crystals. It is also reported that, among single crystals with uniformly deformed orientations, recrystallization takes place most easily in the \(<1\ 1\ 1>\)-oriented crystals (Tagami et al., 2001).

In microscopic studies of deformation and recrystallization of Al single crystals, the experimental methods are primarily based on scanning electron microscopy, e.g., the observation of slip lines on the sample surface, and on crystallographic orientation analysis using electron backscattered diffraction. Transmission electron microscopy has also been applied to observe the dislocation microstructures in deformed samples. However, there had been little effort to non-destructively observe the interior of samples until we first carried out experiments on \(<1\ 1\ 1>\)-oriented Al single crystals using synchrotron X-ray radiation at SPring-8 (Shiro et al., 2013). We found that Al single crystals with fine mosaic structures were imperfect from the crystallographic point of view. In addition, the residual stress evaluated after applying a nominal strain of 8% in the tensile direction was found to be very small compared with the flow stress during deformation.

The major objective of the present study was to carry out a series of observations of the same area in the \(<1\ 1\ 1>\)-oriented Al single crystal at the pre-deformation, post-deformation and post-annealing stages in SPring-8. Almost the whole area of the cross section of the sample was observed. We focused on the effect of sub-boundaries on the deformation and recrystallization behaviors of the sample.

2. Materials and methods

2.1 Samples

Single-crystalline samples were grown by the Bridgman method in a pressure lower than \(6\times10^{-3}\) Pa. The purity of the material was 99.999 mass%. A sample with a cylindrical gauge portion was directly formed in a mold. A single-crystalline seed crystal was used to control the orientation of the sample so that the longitudinal direction was parallel to the \(<1\ 1\ 1>\) direction.

![Photograph of a <111>-oriented Al single crystal prior to tensile deformation.](image)
The grown single-crystalline sample was separated from the seed crystal and annealed in air at 300 °C for 3.6 ks. The sample was then etched with aqua regia. A photograph of the sample is presented in Fig. 1. The central portion had a cylindrical shape, 5 mm in diameter and 20-mm long. We prepared two samples and labeled them samples C and D, following samples A and B used in our previous study (Shiro et al., 2013).

2.2 Experiments using the in-line tensioning/annealing apparatus

We used a specially designed in-line tensioning/annealing apparatus to deform and anneal the sample. Temperature was measured using a thermocouple with a tip located 5-mm from the central portion of the sample. The inside wall of the apparatus was coated with gold to ensure uniformity of temperature.

The nominal strain of 8% in the tensile direction was applied to the sample at room temperature. The strain rate was 3×10⁻⁴/s. We found in previous experiments that a minimum plastic strain of 8% tension was required to recrystallize the <111>-oriented Al single crystal at the annealing temperature of 0.8 T_M (480 °C), where T_M is the melting temperature of Al in the absolute temperature scale. We sought to obtain as large a recrystallized grain as possible by adopting a low tensile strain and high annealing temperature. We knew that the flow stress of 60 MPa corresponded to the 8% plastic strain after unloading. Hence, in the present experiment, the tensile test was carried out until the nominal stress reached 60 MPa.

For sample C, post-deformation annealing was carried out at 480 °C for 900 s. For sample D, after annealing at 480 °C for 150 s, we observed the diffraction spots from the sample using a 2-dimensional (2-D) detector (Pilatus 300K) installed downstream of the beamline, and found that no recrystallized grain was formed at that stage. A second annealing was initiated at 480 °C, and the diffraction spots were monitored. The annealing was terminated when diffraction spots from the deformation matrix disappeared and new diffraction spots appeared. Both samples were heated to 480 °C at a rate of 2.5 °C/s. After the annealing experiments, samples C and D were both allowed to cool naturally to room temperature.

2.3 Diffraction experiments

The diffraction experiments were carried out using the JAEA (Japan Atomic Energy Agency) beamline BL22XU at SPring-8. To map the cross section, the sample was placed on an xyz automated stage mounted on a four-circle goniometer. Since the X-ray beam was collimated by a slit, the size of the irradiated area was estimated to be 0.2×0.173×0.0522 mm³. In this experiment, an X-ray beam of approximately 30 keV was strong enough to pass through the Al sample. The diffracted X-ray beam was detected by an NaI scintillation counter. A schematic of the measurement system is presented in Fig. 2.

![Fig. 2 Measurement system around the four-circle goniometer. The synchrotron radiation (SR) X-ray beam passed through the sample. The diffracted beam made an angle of 2θ with the transmitted beam, where θ is the Bragg angle. The intensity of the diffracted beam was detected by the scintillation counter (SC). The size of incident and diffracted beams was limited by the slits. The φ-, χ-, and ω-rotation of the sample are shown.](image-url)
As may be seen, the rotations of the sample about the tensile axis and the axis parallel to the incident beam are represented as the $\phi$-rotation and $\chi$-rotation, respectively. Since the Bragg angle is taken as $\theta$, the angle between the transmitted beam and diffracted beam is $2\theta$. We irradiated the cross section in the middle of the gauge portion of the sample. The coordinates within the cross section perpendicular to the tensile axis are $\text{stx}$ and $\text{sty}$. Table 1 summarizes the measurement conditions. In the present study, we used diffraction by the (440) plane or (220) plane. Since the tensile axis was parallel to the $<111>$ direction, three $<110>$ axes existed within the cross-sectional plane, and the other three $<110>$ axes were out of plane. Hereafter, the in-plane and out-of-plane $<110>$ axes are referred to as poles 1–3, and 4–6, respectively. Figure 3 contains a schematic of the relationship between the major crystallographic directions.

Table 1 X-ray conditions for the diffraction experiments.

| Parameter                                      | Value                        |
|------------------------------------------------|------------------------------|
| Energy                                         | 29.991 keV                   |
| Monochromatic crystal                          | Si(111)                      |
| Size of divergent slit                         | $0.2H \times 0.05W$ mm$^2$   |
| Size of receiving slit 1                       | $3.0H \times 2.0W$ mm$^2$    |
| Size of receiving slit 2                       | $3.0H \times 0.05W$ mm$^2$   |
| Distance between sample and receiving slit 1   | 275 mm                       |
| Distance between receiving slits 1 and 2       | 1210 mm                      |
| Detector                                       | NaI                          |
| Diffracting plane                              | Al {440} or {220}            |
| Diffraction angle 20                           | $33.57^\circ$ or $16.78^\circ$ |

Fig. 3 Major crystallographic directions of the sample.

The X-ray measurements were carried out in the cross section by changing the positions represented by the values of $\text{stx}$ and $\text{sty}$. At each measured position, after peak refinement by $\phi$-scan, $\chi$-scan, and $2\theta$-scan, a peak profile was obtained by the $\theta$-$2\theta$ scan. The rocking curve obtained by the $\phi$-scan was applicable to the evaluation of crystallinity from the shape and full width half maximum (FWHM) of the profile. The diffraction profile obtained by the $\theta$-$2\theta$ scan was used to calculate the strain from its peak position. Further details regarding the strain-scanning method are described in our previous paper (Shiro et al., 2013). Both samples C and D were measured at three stages: the pre-deformation; post-deformation; and post-annealing stages.
3. Results

3.1 Pre-deformation stage

The values of $2\theta$, $\omega$, $\chi$, and $\phi$ for poles 1–6 measured at the center of the cross section (i.e., $stx = sty = 0$) in samples C and D are presented in Tables 2 and 3, respectively. The values of $2\theta$ and $\omega$ in sample C (Table 2) were double those of sample D (Table 3) because the diffracting planes were {440} and {220} for samples C and D, respectively. Since the deviation of the angle $\chi$ from its ideal value of $0^\circ$ was smaller than $1^\circ$, the orientation control of samples C and D using seed crystals was successful.

Table 2 Values of $2\theta$, $\omega$, $\chi$, and $\phi$ in degrees for poles 1–6 measured at the center of the cross section of sample C at the pre-deformation stage.

| Pole No. | $2\theta$ | $\omega$ | $\chi$ | $\phi$ |
|----------|-----------|----------|--------|--------|
| 1        | 33.56584  | 16.7831  | -0.8   | -35.572|
| 2        | 33.56704  | 16.7835  | -0.4   | 24.431 |
| 3        | 33.56704  | 16.7830  | 0.0    | 84.450 |
| 4        | 33.56524  | 16.7825  | -55.4  | -125.945|
| 5        | 33.56594  | 16.7829  | -55.4  | -4.672 |
| 6        | 33.56494  | 16.7829  | -54.7  | 114.884|

Table 3 Values of $2\theta$, $\omega$, $\chi$, and $\phi$ in degrees for poles 1–6 measured at the center of the cross section of sample D at the pre-deformation stage.

| Pole No. | $2\theta$ | $\omega$ | $\chi$ | $\phi$ |
|----------|-----------|----------|--------|--------|
| 1        | 16.60044  | 8.30022  | -0.4   | 21.5   |
| 2        | 16.60084  | 8.30115  | 0.6    | -37.3  |
| 3        | 16.60094  | 8.30120  | -0.8   | 82.7   |
| 4        | 16.60124  | 8.30135  | -53.9  | -126.5 |
| 5        | 16.60074  | 8.30110  | -54.5  | -8.5   |
| 6        | 16.60114  | 8.30130  | -55.3  | 113.5  |

In order to visualize the distribution of crystallographic orientations within the cross section perpendicular to the tensile axis, measurements were carried out for poles 1–3. We chose these poles because the volume of the irradiated area could be minimized compared to that for poles 4–6, due to the smaller inclination angles of samples for the diffraction experiments. For sample C, measurements at 165 positions at intervals of 0.3 mm were carried out for pole 2 with the smallest $\phi$ angle. The shape, peak position, and FWHM of the rocking curves about the $\phi$ axis differed at measured positions. In other words, the single-crystalline samples used in the present study were not perfect from a crystallographic point of view.

We carried out a Gaussian fitting to determine the peak position and FWHM of the rocking curves. Figure 4(a) is a map of the peak position of pole 2 at each measured position in sample C. Specifically, the peak position at the center of the cross section was taken to be $0^\circ$, and the deviation of the peak position at each measured position was colored. Hence, the map represents the distribution of the crystallographic orientation in the cross section. The largest orientation difference within the cross section was about $1.6^\circ$. It was evident that the orientation distribution was not random, but that the cross section consisted of roughly three regions with different orientation. The boundaries between the regions were clear, with an orientation difference smaller than $1^\circ$, and probably corresponded to sub-boundaries introduced during the crystal growth process. As shown in Fig. 4(b), a similar sub-grain structure was found in sample D, in which the maximum orientation difference within the cross section was about $0.75^\circ$.

Although samples C and D in the present study were annealed after the Bridgman growth process, they were not perfect from the crystallographic perspective, but were composed of several sub-grains. We also carried out orientation mapping of the cross-sectional area of the samples in our previous study, but did not find clear evidence of sub-grain
structure. This was probably because the scanned area was limited to the central portion of the cross section due to the strict temporal restrictions at that time.

3.2 Post-deformation stage

Samples C and D were deformed in tension using the in-line tensioning/annealing apparatus. After unloading, the orientation distribution in the cross section perpendicular to the tensile axis was measured. For sample C, measurements were carried out for pole 2 at 157 positions at 0.3 mm-intervals. Figure 5(a) shows the orientation map constructed from the peak positions obtained from the Gaussian-fitted rocking curves about the \( \omega \)-axis. Specifically, the peak position at the center of the cross section was taken to be 0°, and the deviation angle at each measured position was colored. The color scale of the deviation angle in Fig. 5(a) was set to be the same as that in the pre-deformation stage in Fig. 4(a) for easy comparison. The sub-grain structure in the cross section was similar to that in the pre-deformation stage, while the maximum orientation difference within the cross section increased from 1.6° to 2.3°, or by 0.7°. As mentioned in Section 1, deformation bands are not observed on the surface of Al single crystals deformed in tension along the \(<111>\) direction. The present observation by X-ray diffraction confirmed that there was no deformation band in the interior of the sample.

In sample D, the orientation map of the cross section was constructed by measuring 121 positions at 0.3 mm-intervals. Similar to sample C, the sub-grain structure in Fig. 5(b) did not change much from the pre-deformation stage in Fig. 4(b), while the maximum orientation difference within the cross section increased from 0.75° to 1.4°, or by 0.65°.

Fig. 4 Orientation maps of the cross section of (a) sample C, and (b) sample D at the pre-deformation stage. The orientation differences within the cross section were 1.6° and 0.75° for samples C and D, respectively.

Fig. 5 Orientation maps of the cross section of (a) sample C, and (b) sample D deformed in tension by 8%. The orientation difference within the cross section increased by about 0.7° compared to the pre-deformation stage.
These results clearly showed that the orientation difference between sub-grains increases with tensile strain. The increase in orientation difference induced by an 8% tensile strain was almost the same for samples C and D, i.e., 0.7° and 0.65°, respectively.

As in our previous paper (Shiro et al., 2013), the strain-scanning method was applied to calculate the residual strains in deformed samples C and D. The strains were very small, i.e., $1\times10^{-4}$ and $0.3\times10^{-4}$ for samples C and D, respectively. These values correspond to residual stress of about 10 MPa at most, which is much lower than the flow stress of 60 MPa during tensile deformation. In addition, we determined that the strain distribution had no relation to the sub-boundaries; i.e., there was no locally accumulated strain close to the sub-boundaries.

### 3.3 Post-annealing stage

Post-deformation annealing was carried out for samples C and D using the in-line tensioning/annealing apparatus. Sample C was annealed at 480 °C for 900 s. On the other hand, sample D was first annealed at 480 °C for 150 s. The diffraction spots from the sample were then detected by the 2-D detector installed downstream of the beamline. No evidence of recrystallization was found at that stage. A second annealing was then carried out at the same temperature, and the diffraction spots were monitored. The second annealing was terminated at 432 s, when diffraction spots from the deformation matrix disappeared and new diffraction spots appeared. As a result, sample D was found to recrystallize after annealing at 480 °C for a total of 582 s.

The central portion of samples C and D was completely covered by a single recrystallized grain. Hereafter, we refer to the recrystallized grains in samples C and D as C-R and D-R, respectively. For C-R, in order to visualize the orientation distribution within the cross section perpendicular to the tensile axis, 361 positions were measured at 0.2-mm intervals for pole 2. Unlike the pre-deformation and post-deformation stages, the rocking curve here was very close to the one obtained from an ideal crystal. The peak position of the Gaussian-fitted curve at each measured position was almost the same, and the orientation difference within the cross section was about 0.001°. Therefore, the orientation map constructed by coloring each measured position with the orientation deviation from the center of the cross section was completely uniform, as shown in Fig. 6(a) under the same color scale as in Figs. 4(a) and 5(a). There was also very little orientation variation in the D-R formed in sample D [Fig. 6(b)].

Figure 7 (a and b) shows the crystallographic orientations of recrystallized grains compared to those measured at the center of the samples at the pre-deformation stage using stereographic projections. While the large orientation difference can be clearly observed, there is no evidence of the 40°-rotation relationship about the $<111>$ axes (Yoshida et al., 1959; Senna and Lücke, 1976).
The rocking curve obtained at the center of C-R is presented in Fig. 8(a); it has a symmetrical shape close to a normal distribution curve with FWHM as small as 0.002°. In the figure, the rocking curve from sample C obtained at the pre-deformation stage is included for comparison. The pre-deformation rocking curve is composed of four peaks. As can be seen, FWHM measured after the Gaussian fitting was 12.5 times larger than that in the C-R. It is clear that the crystallinity of the recrystallized grain was far better than that of the Bridgman-grown single crystal. Improved crystal quality was also found for the D-R recrystallized grain shown in Fig. 8(b).

The rocking curve obtained at the center of C-R is presented in Fig. 8(a); it has a symmetrical shape close to a normal distribution curve with FWHM as small as 0.002°. In the figure, the rocking curve from sample C obtained at the pre-deformation stage is included for comparison. The pre-deformation rocking curve is composed of four peaks. As can be seen, FWHM measured after the Gaussian fitting was 12.5 times larger than that in the C-R. It is clear that the crystallinity of the recrystallized grain was far better than that of the Bridgman-grown single crystal. Improved crystal quality was also found for the D-R recrystallized grain shown in Fig. 8(b).

Although the crystallographic qualities of C-R and D-R were almost the same, both being far superior to the as-grown single crystals, there was a slight difference between them. The FWHM values of rocking curves obtained along a straight line at stx = 0 with an interval of 0.2 mm for C-R and D-R are shown in Figs. 9(a) and (b), respectively. It is clear that the fluctuation width of FWHM from C-R [Fig. 9(a)] was smaller than that from D-R [Fig. 9(b)]. This
was probably due to the different annealing processes for deformed samples C and D; i.e., the annealing of sample D was immediately terminated when diffraction spots from the deformation matrix disappeared and were completely replaced by those from a recrystallized grain in the 2-D detector, while sample C was annealed for a prefixed duration. The results show it is possible that the crystallinity of recrystallized grain could be improved by prolonged annealing. We also have to take the difference in the orientations of recrystallized grains in samples C and D into account. A further study is needed to clarify this issue.

4. Discussion

The critical size of the recrystallized grain nucleus is estimated to be about 100 nm in radius, based on the thermodynamic nucleation/growth theory. This corresponds to $10^9$ atoms contained in the nucleus. However, it is very unlikely that such a large number of atoms would be simultaneously activated to form a nucleus during the post-deformation annealing process (Humphreys and Hatherly, 2004a). Hence, it is generally believed that recrystallization nuclei already exist in the deformation matrix. Non-uniformly deformed regions (e.g., deformation bands, grain boundaries, and interfaces with particles or precipitates) play an important role as preferential sites for potential nuclei of recrystallized grains (Humphreys and Hatherly, 2004b).

In the $<111>$-oriented crystals used in the present study, sub-boundaries were clearly visible in the orientation map at the pre-deformation stage. The crystallinity close to the sub-boundaries was virtually unchanged with the application of the tensile strain of 8%; i.e., there was no evidence of local accumulation of dislocations in the regions close to the sub-boundaries. The orientation map of the deformed sample clearly shows that no non-uniformly deformed regions were formed inside the sample. In addition, the level of residual strain/stress calculated by the strain-scanning method was low. Therefore, the sub-boundaries were unlikely to act as preferential sites for recrystallization nuclei. We consider that the potential nucleation sites of recrystallized grains in the $<111>$-oriented crystals are likely located in a small region beyond the resolution of the present experiments. A possible candidate is the intersection of certain types of slip bands (Inoko et al., 2010). The intersection is rotated with respect to the surrounding matrix, and is approximately 0.5 μm in size. Therefore, a sub-micrometer-size beam will be necessary to reveal and further analyze uniformly deformed single crystals, and to explore the potential nuclei of recrystallized grains.

5. Conclusions

$<111>$-oriented Al single crystals were deformed under 8% tension and subsequently annealed. Diffraction experiments using synchrotron X-ray radiation were carried out on the cross section perpendicular to the tensile
direction at the pre-deformation, post-deformation and post-annealing stages. The samples with sub-grain structures were not perfect from the crystallographic point of view, but the orientation difference across the sub-boundaries was smaller than 1°. The 8% tensile deformation induced an increase in orientation difference in the cross section by about 0.7°. However, no evidence was found for the local accumulation of dislocations close to the sub-boundaries. In the annealed samples, the whole cross section was replaced by a single recrystallized grain. The crystallographic quality of the recrystallized grains was far superior to that of the as-grown single crystals. Our findings suggest that the quality of recrystallized grains could be improved by prolonged annealing after recrystallization.

Acknowledgments

The authors are grateful to Mr. Hirotomo Bunya and Mr. Tomoya Kawano at Tokushima University for their technical assistance. The synchrotron radiation experiments were performed at JAEA beamline BL22XU in SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI) (Proposal No. 2013B3723, 2014A3722). This research was partly funded by The Amada Foundation (No. AF-2018003-B2).

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