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***In situ* synchrotron radiation observation of deformation and annealing processes of aluminum single crystal**

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Abstract

The objective of the present study is to carry out *in situ* observations of deformation and annealing of aluminum (Al) single crystals using a synchrotron radiation X-ray at SPring-8. Al single-crystalline samples having a $\langle 111 \rangle$ orientation parallel to the longitudinal direction were grown by the Bridgman method. The samples were deformed in tension to a nominal strain of 8% at room temperature using an in-line tensioning apparatus. Post-deformation annealing at 480 °C was subsequently carried out in the same apparatus. A two-dimensional detector was used to detect multiple diffracted beams from the sample during deformation and annealing. The volume irradiated by the X-ray beam was found to be composed of three regions having small orientation differences attributable to the subgrain microstructures of the sample. Detailed analyses of diffraction spot intensity showed that the subgrain microstructures were surpassed by dislocated microstructures as the tensile strain increased. During post-deformation annealing, diffraction spots from a recrystallized grain first appeared at 180 s after the temperature reached 480 °C. The coexistence of diffraction spots from the deformation matrix and recrystallized grains lasted only about 22 s in the irradiated volume. The migration rate of the boundary between the deformation matrix and the recrystallized grain was estimated to be on the order of several micrometers per second.

Keywords : Aluminum, Single crystal, Tensile deformation, Annealing, Synchrotron radiation

1. Introduction

Static recrystallization has long been studied because of its ability to control the size and crystallographic orientation of grains in polycrystalline materials. Since commercially available alloys often have complex microstructures composed of multiple phases, they are not best suited for fundamental research into the true nature of recrystallization. In addition, practical metal-forming processes such as rolling and drawing are inappropriate for fundamental research because such deformation modes induce complex shear deformation on the sample surface as a result of friction with rollers and dies. To reduce the number of experimental parameters, studies using single crystals of pure metals as samples have been carried out (Ferry and Humphreys, 1996; Stanford et al, 2003; Paul, 2003; Miszczyk et al, 2017). Tensile deformation is preferable for the deformation mode because it is easy to control the number of slip systems involved in the deformation (Yoshida et al, 1959; Senna and Lücke, 1976). Based on this idea, Inoko and coworkers carried out systematic studies on tensile deformation and recrystallization of orientation-controlled aluminum (Al) single crystals (Inoko et al, 1994; Kashihara et al, 1996a, 1996b; Kashihara 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).

Al single crystals deformed in tension can be classified into two groups, depending on whether or not the crystals

are deformed by deformation bands. For example, when the tensile axis is parallel to the $\langle 110 \rangle$ or $\langle 1\ 4\ 10 \rangle$ direction, deformation bands are formed (Inoko et al, 1994; Kashiwara et al, 1996a, 1996b; Okada et al, 2001; Wert et al, 2003; Okada et al, 2003). Post-deformation annealing induces the formation of recrystallized grains along the boundaries between the deformation matrix and deformation band. On the other hand, when Al single crystals are deformed in tension along the $\langle 111 \rangle$, $\langle 112 \rangle$ or $\langle 001 \rangle$ direction, multiple slip systems operate from the initial stage of deformation and macroscopically uniform deformation proceeds without the formation of deformation bands (Kashiwara et al, 2000; Tagami et al, 2001). Work-hardening behavior among these uniformly-deformed orientations is totally different, depending on the ease or difficulty in cross slip. Al single crystals deformed in tension along the $\langle 111 \rangle$ direction (hereafter, “ $\langle 111 \rangle$ -oriented crystals”) show the largest work-hardening due to the complete suppression of large-scale cross slip. Heavily accumulated dislocations in the $\langle 111 \rangle$ -oriented single crystals lead to relatively easy recrystallization by post-deformation annealing (Tagami et al, 2001).

Previous recrystallization studies employing Al single crystals were based mostly on surface observation and orientation analysis by scanning electron microscopy and electron back-scattered diffraction analysis, respectively. To observe the interior of the samples, destructive observation with transmission electron microscopy was necessary. We first carried out non-destructive interior observation of Al $\langle 111 \rangle$ -oriented crystals using synchrotron radiation X-ray at SPring-8 (Shiro et al, 2013) and found that the Al crystals were not perfect but rather were composed of several subgrains with small orientation differences, which could not be removed by pre-deformation annealing. The samples were deformed in tension and subsequently annealed to evaluate the crystallinity of deformed microstructures and recrystallized grains (Shiro et al, 2020).

In those studies, a scintillation counter was used to detect a diffracted X-ray beam. This device is a powerful tool for precisely measuring an irradiated point, and crystallographic orientation maps across the entire cross section of the sample at the pre-deformation, deformation and recrystallization stages were constructed by repeating the point measurements. However, since the mapping is very time-consuming, it cannot be applicable to *in situ* observation. In the present study, we used a two-dimensional (2D) detector to detect multiple diffracted beams from the sample during deformation and annealing. We successfully observed the transition from the deformed state to recrystallization during annealing, and were able to estimate the migration rate of the boundary between the deformation matrix and recrystallized grain in the interior of the sample.

2. Experimental procedures

2.1 Samples

A modified Bridgman method was used to grow Al single-crystalline samples. The purity of the material was 99.999 mass%. Seed crystals with a rectangular parallelepiped shape were prepared from a bulk single crystal so that the longitudinal direction of the seed crystal was parallel to the $\langle 111 \rangle$ direction. The seed crystal and an Al ingot were placed at the lower and upper positions in a graphite mold, respectively. Crystal growth was carried out in a vacuum with a pressure below 6×10^{-3} Pa. After the ingot and the upper portion of the seed crystal were melted, the furnace was slowly moved upwards so that a single crystalline sample with a cylindrical gauge portion (5 mm in diameter and 20 mm in length) was formed directly in the mold. The successfully grown sample had the same orientation as the seed crystal. After the sample was separated from the seed crystal, pre-deformation annealing was carried out at 300 °C for 3.6 ks in air. The surface of the annealed sample was etched with aqua regia. Two samples were prepared and labeled D and E. Sample D was the same as sample D in our previous paper (Shiro et al, 2020) in which we reported on microstructural analyses at the pre-deformation, post-deformation and post-annealing stages of the sample. In the present paper we report on the *in situ* observation of the intensity change in a diffraction spot during tensile deformation. Sample E was newly prepared for the present study.

2.2 Experiments using tensioning/annealing apparatus

Tensile testing and post-deformation annealing were carried out using an in-line tensioning/annealing apparatus. The strain rate of the tensile test was 3×10^{-4} /s, and 8% strain was applied to the samples. Sample D was used to measure the intensity change of a 400 diffraction spot during the tensile test at room temperature. Sample E was similarly deformed to a tensile strain of 8%. After unloading, post-deformation annealing was carried out in the same apparatus. The temperature was measured by a thermocouple whose tip was placed 5 mm from the center of the gauge

portion of the sample. The inside wall of the apparatus was coated with gold to ensure uniformity of temperature. The temperature was raised from room temperature to 480 °C in 194 s and maintained for 900 s, followed by natural cooling to room temperature in about 1.2 ks.

2.3 *In situ* synchrotron radiation measurements

In situ synchrotron radiation measurements were carried out at the Japan Atomic Energy Agency (JAEA) beamline BL22XU at SPring-8. A synchrotron X-ray beam with an energy of 29.991 keV (wavelength: 0.041339 nm) was focused at the center of the gauge portion of the sample. The area irradiated by the X-ray beam was limited by a single slit of 0.2 mm in height and 0.05 mm in width as schematically shown in Fig 1 (a). Since the energy of the X-ray was high enough to penetrate through the sample, both transmitted and diffracted beams were generated at the downstream side of the sample, as schematically shown in Fig. 1 (b). The angle between the transmitted and diffracted X-ray beams is 2θ , where θ is the Bragg angle of the diffracting plane. To detect the diffracted X-ray beams, a 2D detector (Pilatus 300K) was installed at a camera length of 170 mm from the sample. Due to the limited size of the detecting plane, X-ray beams diffracted by the planes having interplanar spacing between the 220 and 440 lattice planes were detected. Since the samples used in the present study were single crystals, discrete diffraction spots were generated. To detect as many diffraction spots as possible, a swing motion was applied to the sample as schematically shown in Fig. 1 (b). The swing motion was in an angular range of $\pm 15^\circ$ and the angular velocity was $1^\circ/\text{s}$. Therefore, the period of a single swing motion was 30 s. Three-hundred images taken at a frequency of 10 Hz during the swing period of 30 s were combined into a single image to show the distribution of diffraction spots from the sample.

Fig. 1 (a) Schematic of sample viewed from the upstream side of X-ray. The irradiated area is at the center of the gauge portion, 0.2 mm in height and 0.05 mm in width. “Sun” and “Earth” are used to represent the gravity direction. (b) Arrangement of sample and 2D detector. A diffracted X-ray beam is generated from a plane that satisfies the Bragg condition, making an angle of 2θ with the transmitted X-ray beam, where θ is the Bragg angle. The 2D detector is installed at a camera length of 170 mm from the sample. To detect as many diffraction spots as possible from the single-crystalline sample, ω -swing motion is applied to the sample. The x- and y-axes in front of the 2D detector show the coordinate axes of the detecting plane. T.A. stands for the tensile axis of the sample, parallel to (111).

3. Results

3.1 Deformation process

The stress-strain curve of sample D is presented in Fig. 2. In the graph, the ordinate is the nominal tensile stress obtained by dividing the load by the initial cross-sectional area of the gauge portion of the sample. The strain in the abscissa was determined by assuming that the maximum crosshead displacement of the tensioning apparatus corresponded to the tensile strain of 8%. The stress monotonically increases, reaching 60 MPa at the maximum strain. The overall shape of this stress-strain curve agrees well with that reported previously (Tagami et al, 2001).

To evaluate the dynamic change in the diffraction spot during tensile deformation, the image intensity of a diffraction spot obtained by the 2D detector was integrated and converted into one-dimensional data. The results for a 400 spot are presented in Fig. 3. Each profile of the 400 diffraction from a tensile strain of 0.038% to 8.27% is drawn in the figure, where the strains were determined by simply dividing the crosshead displacement by the initial gauge length of the sample. In the figure, the abscissa corresponds to the positions along the x-axis of the detecting plane (see Fig. 1 (b)) measured by pixels. From a perfect single crystal, a sharp profile with a single peak should appear. However, it is

remarkable that profiles within 1.08% are composed of three separate peaks as shown in Fig. 3. This indicates that the sample was not perfectly single-crystalline but that there were three regions with slightly different crystallographic orientations in the volume irradiated by the X-ray beam. From our previous study, it is known that Al crystals grown by the Bridgman method are not perfect but rather are composed of several subgrains with small orientation differences, which cannot be removed by pre-deformation annealing (Shiro et al, 2020). The intensity profiles of a diffraction spot with multiple peaks in Fig. 3 reflect the subgrain microstructures of the sample.

Fig. 2 Stress-strain curve for sample D. The tensile stress on the ordinate is the nominal stress. The strain on the abscissa was determined by assuming that the maximum crosshead displacement of the tensioning apparatus corresponded to the tensile strain of 8%.

Fig. 3 Dynamic change in diffraction intensity of a 400 spot from sample D during tensile deformation. Profiles were obtained for tensile strains between 0.038% and 8.27% by integrating the intensity of the spot image. The x position on the abscissa corresponds to that on the detecting plane (see Fig. 1 (b)).

One pixel along the abscissa in Fig. 3 corresponds to 0.172 mm on the 2D detector plane. From this relationship, we can estimate the orientation difference $\Delta\theta$ between two prominent peaks separated by seven pixels (1.204 mm) at the strain of 0.038% in Fig. 3. From the geometry of Fig. 1 (b), we derive the following Eq. (1), where the camera length $L = 170$ mm, the Bragg angle $\theta = 11.78^\circ$ for the 400 lattice plane and the distance $p = 1.204$ mm between the two prominent peaks. The obtained value $\Delta\theta = 0.17^\circ$ is smaller than the maximum orientation difference of 0.75° within the entire cross section of sample D (Shiro et al, 2020). This difference is accounted for by the smaller size irradiated by the X-ray beam compared to the entire cross section.

$$L [\tan 2(\theta + \Delta\theta) - \tan 2\theta] = p \quad (1)$$

At the initial stage of deformation, i.e., for strains lower than 1.08%, the intensity of the diffraction spot decreased little, but only a small shift in the peak positions was observed. This suggests that, although dislocation slip occurred, the accumulation of dislocations was not large at that stage. With the increase in the tensile strain, the intensity decreased significantly and prominent peaks disappeared from the profile. For strains higher than 4%, the intensity profiles became featureless, and the development of dislocated microstructures in the sample surpassed the effect of the subgrain microstructures.

3.2 Annealing process

Sample E was deformed to a tensile strain of 8% and subsequently annealed in the tensioning/annealing apparatus. As described in Section 2.3, the images taken by the 2D detector were obtained continuously at a frequency of 10 Hz, swinging the sample at the angular velocity of $1^\circ/\text{s}$ in the angular range of $\pm 15^\circ$. The images taken during a period of the swing motion were combined into a single image as presented in Fig. 4. The x - and y -directions in the figure correspond to the x - and y -directions in front of the detecting plane as drawn in Fig. 1 (b). The arcuate dotted lines in Fig. 4 represent the positions on which diffraction spots should appear. Figures 4 (a), (b) and (c) are the combined images for the periods of 336-366 s, 366-396 s and 396-426 s, respectively. In the figures, diffraction spots from the deformation matrix and the recrystallized grain are distinguished using orange and blue circles, respectively. In Fig. 4 (a), diffraction spots appear only from the deformation matrix. As a result of the orientation variation in the deformation matrix, the diffraction spots in Fig. 4 (a) are relatively large. Diffraction spots from the deformation matrix and the recrystallized grain coexist in Fig. 4 (b). Compared to the diffraction spots from the deformation matrix, those from the recrystallized grain were smaller due to the high crystallinity of recrystallized grains (Shiro et al, 2020). By checking the 300 images taken during the period of 366-396 s, we found that diffraction spots from the recrystallized grain first appeared at 374 s, i.e., 180 s after the temperature reached 480°C . In Fig. 4 (c), diffraction spots from the deformation matrix disappeared completely. We observed diffraction spots only from the recrystallized grain. The coexistence of the deformation matrix and the recrystallized grain lasted for 22 s, i.e., from 374 s to 396 s. In other words, the boundary between the deformation matrix and the recrystallized grain passed through the region irradiated by the X-ray beam in 22 s. The migration rate of the boundary during the recrystallization was at least $2.3\ \mu\text{m}/\text{s}$, which was obtained by simply dividing 0.05 mm by 22 s. Here, 0.05 mm is the shorter side of the area irradiated by the X-ray. Hence, the migration rate is roughly estimated to be on the order of several micrometers per second.

The elongated shape of diffraction spots from the recrystallized grain is attributable to the thickness of the sample. In the present study, a single slit was installed on the upstream side of the sample, and diffraction through the sample thickness was integrated. As a result, the larger the diffraction angle 2θ , the more elongated the diffraction spots.

Fig. 4 The images taken by the 2D detector during the post-deformation annealing of sample E were combined into a single image. Panels (a), (b) and (c) were taken during the ω -swing periods of 336-366 s, 366-396 s and 396-426 s, respectively. The arcuate dotted lines represent the positions on which diffraction spots should appear. Diffraction spots from the deformation matrix and the recrystallized grain are distinguished by the colors of the circles.

In order to discuss the linear expansion coefficient of the recrystallized grain, the diffraction spot R in Fig. 4 (b) was selected, which corresponds to a 220 diffraction from the recrystallized grain. In the discussion, diffraction data were reconstructed on the plane of 2θ - and ω -angles. First, an image of a diffraction spot taken by the 2D detector at an ω -angle was converted into one-dimensional data by integration. This procedure was repeated for other ω -angles. Finally, the obtained data were plotted on the 2θ - ω plane as shown in Figs. 5 (a) and (b). In the figures, the integrated diffraction intensity was represented by a color code with contour lines, where the color key accompanies each figure. The 2θ - and ω -angles in the abscissa and ordinate represent the spacing of diffracting planes and the crystallographic orientation, respectively. Figure 5 (a) is reconstructed from the 220 spot R in Fig. 4 (b). Figure 5 (b) is from the same spot as Fig. 5 (a) but the data were reconstructed after the sample was cooled to room temperature, 20 °C. The overall shape and size of the reconstructed data from the 220 diffraction from the recrystallized grain are almost the same at high temperature as at room temperature, i.e., Figs. 5 (a) and (b). However, the position of the intensity maximum was shifted by about 0.12° toward the higher angle by cooling from 480 °C to 20 °C. This was because the interplanar spacing became narrower as the temperature decreased. From these data we can estimate the linear expansion coefficient along the [110] direction which is perpendicular to the 220 lattice planes. Here we take the interplanar spacing of the 220 lattice planes at room temperature (20 °C) as 0.14317 nm. The Bragg angle for 220 lattice planes at room temperature is 8.30°. By calculating the interplanar spacing of the 220 lattice planes at 480 °C from Bragg's law, we can estimate the linear expansion coefficient along the [110] direction as 15.7×10^{-6} /K, which is comparable to the linear expansion coefficient, 26.5×10^{-6} /K, obtained for Al polycrystals.

The high intensity regions in Fig. 5 have 2θ widths of approximately 0.5°. This is attributable to the effect of sample thickness. As described previously, diffraction from each point along the sample thickness is integrated. In Fig. 5, the difference in camera length from each point to the 2D detector is converted into the difference in the 2θ angle. Since the sample thickness is the same, we obtain the same 2θ width for all diffraction spots.

Fig. 5 The diffraction data were reconstructed on the plane of 2θ - and ω -angles. The intensity obtained from integration is represented by the color code with contour lines. Panels (a) and (b) were reconstructed from spot R in Fig. 4 (b). Panel (b) was reconstructed from the same spot as panel (a) after cooled to room temperature.

4. Discussion

Experimental measurements of the mobility of high-angle boundaries during recrystallization are limited compared to those of curvature-driven grain growth. Huang and Humphreys (1999) reported on the grain boundary mobility during recrystallization of an Al-0.05%Si single phase alloy. In their measurements using *in situ* annealing in a scanning electron microscope, the boundary migration rates between 0.1-0.14 $\mu\text{m/s}$ were measured for a 30% cold-rolled sample during the post-deformation annealing at 315 °C. This value is approximately 20 times smaller than that estimated in the present study (2.3 $\mu\text{m/s}$). However, this difference is reasonably explained by taking into account that the grain boundary mobility M is temperature dependent and is usually found to obey an Arrhenius type relationship, Eq. (2),

$$M = M_0 \exp (-Q/RT) \quad (2)$$

where M_0 is a constant, T the absolute temperature, Q the activation energy for boundary migration and R is the gas constant. Using the activation energy of 67 kJ/mol for boundary migration in Al (Humphreys and Hatherly (2004)), the mobility at 480 °C is calculated to be 20 times larger than that at 315 °C. To relate the boundary mobility to the boundary migration rate, we also have to estimate the driving pressure which generally arises from a stored dislocations in the sample. Further study is necessary to evaluate the dislocation density in the interior of deformed samples using synchrotron radiation.

5. Conclusions

Synchrotron radiation X-ray diffraction was used for *in situ* observations of tensile deformation up to 8% strain and subsequent annealing at 480 °C of $\langle 111 \rangle$ -oriented Al single-crystalline samples. A swing motion was applied to a sample to detect as many diffracted X-ray beams as possible by a 2D detector. The samples were not perfectly single-crystalline but rather composed of three subgrains having an orientation difference of 0.17° in the volume irradiated by the X-ray beam. This feature became indistinct as the tensile strain exceeded 4% probably because the effect of dislocated microstructures surpassed that of the subgrain microstructures. During the post-deformation annealing, the formation of a recrystallized grain was first detected at 180 s after the temperature reached 480 °C. In the volume irradiated by the X-ray, the deformation matrix and the recrystallized grain coexisted for 22 s, after which diffraction spots from the deformation matrix disappeared completely. From this observation, the migration rate of the boundary between the deformation matrix and the recrystallized grain was estimated to be on the order of several micrometers per second.

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