Effect of Cross Slips on Deformation Microstructure and Recrystallization in (111) and (001) Al Single Crystals

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(111) and (001) aluminum single crystal specimens with 99.99 mass% purity were deformed in tension to strains of about 20%. In all specimens, multiple slip structures without deformation bands were observed. In (111) specimen deformed at room temperature of 293 K (RT), fine wavy slip structures are recognized because of the difficulty of cross slips. The difficulty is due to the tensile-orientation dependence of cross slips. The dislocation structure shows layered cell structures composed of cell walls with high dislocation density. In (001) specimen deformed at liquid nitrogen temperature of 77 K (LNT), complex fine slip traces similar to those in the case of (111) RT specimen are also observed because of the difficulty of cross slips. This difficulty is due to the temperature dependence of cross slips. The dislocation structure is composed of small isotropic cells with high dislocation density around their cell walls. In the above two kinds of deformed aluminum single crystals, the formation of recrystallized grains (RGs) is very easy. On the other hand, in the (001) specimen deformed at RT, many cross slips with large steps are seen because all the eight primary slip systems have an appropriate cross (i.e. another primary) slip system geometrically. The dislocation structure gives polygonal cells with low dislocation density. After annealing no recrystallized grain is formed in the specimen. The stress values of the stress-strain curves in the (001) (RT), (111) (RT) and (001) (LNT) specimens are 22 MPa at 25% strain, 71 MPa at 22% and 106 MPa at 20%, respectively.

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1. Introduction

In general, deformation bands (DBs) can be observed in deformation structures not only in single crystals,¹⁻⁶ but also in polycrystals of metals.¹⁰ Honeycombe¹¹ and Takamura¹² classified such deformation bands into two kinds, that is, kink bands (KBs) and bands of secondary slip (BSSs). It is noted that a KB crosses normally primary slip bands while a BSS forms in the direction parallel to those.²³ Inoko et al.⁵,⁶ studied KBs in aluminum single crystals deformed in tension to strains of 0.3. Figure 1 indicates tensile orientations of Schmid factor of 0.5, (011), (001), (112) and (111) in a fundamental triangle of (001) – (011) – (111) by a stereographic projection. We have to notify how to determine the primary, critical, conjugate and cross slip planes. The primary slip plane “P1” shown in Fig. 1 was named to the slip plane with the maximum value of Schmid factor. And P2, P3 and P4 were labeled the critical, conjugate and cross slip planes, respectively, based on Rosi and Mathewson’s criterion.¹¹ The six slip directions were called D1 to D6. P1 means the inverse slip plane of P1, and D1 the inverse direction of D1. The symbol “P1D1” means the primary slip system of the plane P1 and the slip direction of D1. Figure 2 shows a schematic diagram of a KB in an aluminum single crystal with a tensile orientation of Schmid factor of 0.5, deformed in tension to a strain of 0.3. The KB crosses primary slip bands of P1D1 at an angle of 60° after a strain of 0.3. In the KB, slip bands of the critical slip plane “P2” are observed remarkably. A pair of high angle pseudo-boundaries is formed between the KB and its adjoining deformed matrixes. The primary slip bands of P1D1 in the KB(P1D1(DB)) incline at 30° counterclock-

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wise for those in the DM(P1D1(DM)). Therefore, it should be noted that in the KB there exists compressive strain due to the operation of critical slip systems P2D2 and P2D5 even during tensile deformation. In aluminum single crystal deformed in tension for a direction of (011), two kinds of bands are formed alternately. One corresponds to region slipped by primary slip systems of P1D1 and P1D3, and the other corresponds to region slipped by the other primary slip systems P2D2 and P2D5. Kashihara et al. called these bands “special type of bands of secondary slips (SBSS)”.

When these aluminum specimens deformed in tension by less than 50% strain are annealed, recrystallized grains (RGs) are formed along the DBs, and many RGs have the (111) rotation relationships to the DBs or the DMs. Figure 3 gives a model of the (111) rotation relationship between the RGs (nuclei) and the DBs or DMs. For example, a RG(cylindrical nucleus) rotated about an axis normal to P4(cross slip plane) is given by networking by two kinds of dislocation loops, that is, P1D1 and P2D5, around itself. In the specimens deformed to more than 50% strain, RGs having the strain induced grain boundary migration (SIBM) are predominant along the original DBs together with the RGs having the (111) rotation relationships. These results indicate unambiguously that DBs such as KBs and SBSSs become preferential nucleation sites for the RGs having the (111) rotation relationships and the SIBM.

Single crystal specimens with stable tensile axes such as (001), (112) and (111) are expected to develop multiple slip structures without formation of DBs, whose misorientations are much smaller than those between the DBs and the DMs. Therefore, the formation mechanism of RGs nucleated from the multiple slip structures is expected to be useful for control of recrystallization texture.

In the present study, two aluminum single crystals with tensile axes of (001) are deformed in tension at room temperature (RT) and liquid nitrogen temperature (LNT), respectively. Hereafter they are called “(001) RT specimen” and “(001) LNT specimen”, respectively. A single crystal with a tensile axis of (111) is deformed at RT (hereafter called “(111) RT specimen”). Their multiple slip structures are investigated to clarify tensile orientation and deformation temperature dependence of cross slips. And the effect of cross slips on the formation of RGs is discussed examining deformed and recrystallized structures in detail.

2. Experimental Procedure

99.99 mass% aluminum single crystals with tensile axes of (001) and (111) were prepared by a soft-mold Bridgman technique. From them, (001) and (111) specimens for tensile tests were cut by an electrical discharge machine. Their surfaces were mechanically polished with emery papers and electrolytically polished with a 20% ethanol +80% perchloric acid solution in volume. The observed planes of (001) and (111) specimens were [011] and [112], respectively. Figure 4 shows their initial orientations. Table 1 indicates the values of Schmid factor for twelve slip systems in (001) and (111) specimens. The primary slip plane “P1” given in Fig. 4 was named to the slip plane actually most activated in deformation. The sizes of parallel parts of the test specimens were 7 mm 4 mm 10 mm for (001) RT and LNT specimens and 4.5 mm 4 mm 10 mm for (111) RT specimen. These specimens were annealed at 773 K for 3600 s before the tensile tests to remove residual strain. (111)RT specimen was deformed in tension to a strain of 22% at a strain rate of 3 x 10^-4 s^-1 at RT. (001) RT specimen was deformed to 25% at RT and (001) LNT one to 20% at LNT. The deformed structures on the specimen surfaces were observed using a scanning electron microscope (SEM), and the orienta-
Table 1 Schmid factor of slip systems in (111) and (001) specimens.

<table>
<thead>
<tr>
<th>Slip systems</th>
<th>Tensile direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1D1</td>
<td>(001)</td>
</tr>
<tr>
<td>P1D3</td>
<td>0.45</td>
</tr>
<tr>
<td>P1D4</td>
<td>0.04</td>
</tr>
<tr>
<td>P2D2</td>
<td>0.46</td>
</tr>
<tr>
<td>P2D4</td>
<td>0.43</td>
</tr>
<tr>
<td>P2D5</td>
<td>0.02</td>
</tr>
<tr>
<td>P3D5</td>
<td>0.40</td>
</tr>
<tr>
<td>P3D3</td>
<td>0.04</td>
</tr>
<tr>
<td>P3D6</td>
<td>0.43</td>
</tr>
<tr>
<td>P4D1</td>
<td>0.40</td>
</tr>
<tr>
<td>P4D5</td>
<td>0.02</td>
</tr>
<tr>
<td>P4D6</td>
<td>0.42</td>
</tr>
</tbody>
</table>

The dislocation structures after the deformation in (001) RT and LNT specimens, and (111) RT specimen were observed using a transmission electron microscope (TEM), and the orientations at local areas were analyzed by electron diffraction patterns (EDPs). The three specimens deformed in each condition were annealed to obtain initial stage of primary recrystallization by changing heating temperatures and annealing times. The recrystallized structures were observed using the SEM to count the numbers of RGs. And then the orientation relationships between the RGs and the neighboring DMs were analyzed from their ECPs.

3. Results and Discussion

3.1 Stress-strain curves and deformation microstructures

3.1.1 Stress-strain curves

Figure 5 shows stress-strain curves of (001) RT and LNT specimens, and (111) RT specimen. The stress value in (001) RT specimen was almost saturated even at 2% strain. The stress value at 25% strain was 22 MPa. The rate of work-hardening in (111) RT specimen was always much larger than that of (001) RT specimen. The value of stress at 22% strain was 71 MPa, which was three times higher than that of (001) RT specimen. The rate of work-hardening in (001) LNT specimen was the largest in the measured strains. The value of stress at 20% strain was 106 MPa, which was five times higher than that of (001) RT specimen.

3.1.2 SEM observations of deformation microstructures

The deviations of actual tensile axes from the (111) axis in the specimens were within 4 degrees.

Figure 6 shows a SEM micrograph imaging slip bands in (111) RT specimen. A lot of fine slips were recognized almost parallel to P1 or P2, and the shape of fine slip bands was wavy and most likely composed of the slip bands of P1 (or P2), P3 and P4. Some of long slip bands parallel to P3 and P4 were also seen, but no cross slip took place macroscopically on the slip systems. The maximum misorientation of the deformed structure was within several degrees by the measure of ECPs.

Figure 7 shows a SEM micrograph of slip bands in (001) RT specimen. Large steps of cross slips were recognized,
which were formed at the initial stage of deformation.\textsuperscript{12} They were cross slipped from P1 (or P3) to P2 and P4, and vice versa. Small steps of cross slips were also observed, which developed at the later stage of deformation. In (001) LNT specimen shown in Fig. 8, complex and fine slip bands were observed. Their directions did not correspond to exactly slip bands of P1 (or P3), P2 and P4 determined from the orientation measurement. Long straight slip bands parallel to these slip planes were recognized. It was found that the cross slips were suppressed at least macroscopically in the tensile deformation at the low temperature, namely at LNT. The formation of DB was not observed in all the specimens, and each maximum misorientation in their deformation structures was within several degrees.

3.1.3 TEM observations of deformation microstructures

Figure 9 shows a TEM micrograph of dislocation substructure in (111) RT specimen. A typical layered structure was developed. In particular, cell walls with high dislocation density parallel to P1 or P2 were observed. Thin cell walls were also formed in directions parallel to P3 and P4. A TEM micrograph of dislocation substructure in (001) RT specimen was given in Fig. 10. It should be noted that isotropic cell structure composed of cell walls with low dislocation density was observed. Figure 11 shows a TEM micrograph of dislocation substructure in (001) LNT specimen. In this case, isotropic cells with high dislocation density were developed. The size of cells in (001) RT specimen was much larger than that in (001) LNT specimen.

The orientations of the dislocation substructures in (111) RT specimen, and (001) RT and LNT specimens were measured with EDPs. In result, the maximum misorientations in the deformation substructures of all the specimens were within several degrees. In order to observe the dislocation substructures in more detail, TEM micrographs were taken from $g = (111)$ parallel to the tensile axis of (111) specimen, and $g = (002)$ parallel to those of both (001) specimens. The magnified substructure in (111) RT specimen was shown in Fig. 12. Many dislocations were recognized in the layered cell walls, but there were almost no dislocations in the interiors of cells. Figure 13 shows the magnified substructure in (001) RT specimen. The polygonal cell walls with low dislocation density were seen, whose characteristic would be influenced
Fig. 12. TEM microstructure of (111) RT specimen deformed in tension to a strain of 22%.

Fig. 13. TEM microstructure of (001) RT specimen deformed in tension to a strain of 25%.

Fig. 14. TEM microstructure of (001) LNT specimen deformed in tension to a strain of 20%.

Fig. 15. (a) Contour maps of Schmid factor of cross slip system, and (b) the ratio of Schmid factor for cross slip system to that of primary slip one (from Diel et al.\textsuperscript{13}).

strongly by the cross slips. The structure in (001) LNT specimen shown in Fig. 14 was composed of polygonal or round cell walls with high dislocation density. A lot of dislocations were tangled each other even in the cell walls.

3.1.4 Relationship between the stress-strain curves and the dislocation substructures

Figure 15 shows two contour maps for the value of Schmid factor of a cross slip system and the ratio of Schmid factor of the cross slip system to that of the primary slip system represented by Diel et al.\textsuperscript{13} and Fujita et al.\textsuperscript{14}. As shown in Fig. 15(b), the ratio becomes positive in the triangle of (011) – (112) – (001), whereas negative in that of (011) – (112) – (111). On the line of (011) – (112), the ratio is always ±0. The cross slip can take place easily at the tensile axis of (001) with the ratio of +1, whereas the occurrence of cross slip is geometrically difficult at (111) axis with the ratio of −1. This fact means that the cross slip from P1D1 to P4D1 is very difficult to take place in (111) specimen. This prediction agrees well with the actual results obtained in the present study. Namely the cross slips were observed very easily in (001) RT specimen, whereas not in (111) RT specimen at least macroscopically.

Mura et al.\textsuperscript{13} reported that angle between the cross slip plane and the primary slip plane determines the occurrence of cross slip. In the case of (111) specimen in which the angle between them is acute, the cross slip does not occur. In contrast, the angle in (001) specimen is obtuse, so the cross slips take place easily. Here, three-dimensional models on the angle relation between the primary slip system to the cross slip system are represented in Fig. 16. In (111) RT specimen, when the cross slips take place from P1 to P4, the primary slip direction is D1 on the plane of P1 whose normal is upward, whereas the cross slip direction is D1 on P4 whose normal is downward. So, since operation of P4D1 produces tensile strain, the cross slip system of P4D1 should undergo compressive strain geometrically. Therefore, at least in a macroscopic scale, it is considered that the occurrence of cross slip
from P1D1 to P4D1 in (111) RT specimen is difficult. On the other hand, in the case of (001) specimens, both slip systems P1D1 and P4D1 with the high values of Schmid factor as the primary slip systems can operate in the same direction of D1, because the normals to P1 and P4 are upward. Therefore, the cross slips from P1D1 to P4D1 and vice versa in (001) RT specimen occur very easily. The cross slips from P1D4 to P2D4, P2D2 to P3D2 and P3D6 to P4D6 are also very easy because of the appropriate geometric relations for the cross slips similar to the combination of P1D1 and P4D1.

Now, let us compare the microstructures observed in SEM (Figs. 6, 7 and 8) and TEM (Figs. 9, 10 and 11) with their stress-strain curves shown in Fig. 5. (001) RT specimen, in which a lot of cross slips occurred, was characterized by such stress-strain curve that the stress was saturated almost at 2% strain and the rate of work-hardening was close to zero. The value of stress at 25% strain was only 22 MPa. The saturation would be caused by the occurrence of cross slips to avoid the storage of dislocations. At the initial stage of deformation, since there was low density of the stored dislocations, large steps of the cross slips were observed. But, the steps of cross slips became short with increasing the tensile strain.\(^\text{12}\)

The flow stress in (111) RT specimen was increased with strain over 20% strain. The value of stress at 22% strain was 71 MPa, which was almost three times higher than that in (001) RT specimen. This high value of stress in (111) RT specimen is caused by the difficulty of cross slips. So, the deformation in (111) RT specimen had to proceed only by the fine multiple slips. However, there is still a possibility that cross slips occur in very short range due to piled-up dislocations although the compressive strain occurs geometrically by cross-slipping from the primary slip systems even during tensile deformation.

The value of stress at 20% strain in (001) LNT specimen was 106 MPa, which was about five times higher than that in (001) RT specimen. The complex and fine multiple slips were introduced to avoid the pre-existing dislocations acted as a barrier. The dislocations introduced in the deformation substructure were tangled with each other, because of the suppression of cross slips in the deformation at LNT. So, the flow stress was increased with the increase of strain in contrast with that in (001) RT specimen.

3.2 Recrystallization

Figure 17 shows a schematic diagram of annealing conditions for recrystallization in (111) RT specimen, and (001) RT and LNT specimens. All deformed specimens were put into an electric furnace keeping the temperature of 753 K. After annealing every 2 or 3 minutes the specimens were taken out of the furnace, and then the states of recrystallization were checked. Both (111) RT and (001) LNT specimens were recrystallized after the annealing at 753 K for 930 s. However, (001) RT specimen was not recrystallized even at higher temperatures and for longer annealing times of 753 K-2850 s, 803 K-180 s, 833 K-180 s, 873 K-180 s and 903 K-480 s. It is reasonable to note that since as shown in Fig. 10 dislocation density is low in (001) RT specimen any recrystallization nucleus is not formed. So, this behavior would correspond to “in-situ recrystallization”.

Figure 18 shows the recrystallized structure and its schematic illustration in (111) RT specimen, and the (111) pole figure of the initial orientation, the deformed structure and the RGs. Fifteen of twenty RGs were characterized by the (111) rotation relationships to the DMs. The RGs rotated about the axes normal to P1 and P2 planes were three in number, respectively. The numbers of RGs belonging the rotation about the axes normal to P3 and P4 were two and eight, respectively. The rotation angles of the RGs with the (111) rotation relationships were 36 to 40 degrees clockwise and 26 to 50 degrees counterclockwise. It means that the formation of RGs in the present study is not explained by the coalescence theory\(^\text{15,16}\) or the SIBM theory\(^\text{10,17,18}\) but by the other nucleation theory described elsewhere,\(^\text{19,20}\) in which the RGs originate at the intersections of cell walls or the slip bands undergo heavy shear strain.

Next, we have to know the reason why many RGs with the rotation axis normal to P4 were observed in (111) RT specimen. It is considered that screw dislocation networks composed of P1D1 and P3D6 (P2D5 in Fig. 3) were more frequently formed than the other networks, which could produce the RGs rotated about the axis normal to P4, based on the (111) rotation recrystallization model proposed by Inoko \textit{et al.}\(^\text{6,19}\) and Kashihara \textit{et al.}\(^\text{21}\) Both P1D1 and P3D6 would be most activated in (111) RT specimen. It mainly depends on the values of Schmid factor shown in Table 1 and is shown in
the SEM micrograph of Fig. 6.

The recrystallized structure and its schematic illustration in (001) LNT specimen, and the orientation relationships between the RGs and the DMs were given in Fig. 19. Four of six RGs had the (111) rotation relationships to the DM. One RG with the rotation axis normal to P1 was rotated by 30 degrees counterclockwise to the DM. The number of RGs with the rotation axis normal to P2 was two, whose rotation angles were 15 degrees clockwise and 20 degrees counterclockwise, respectively. One RG with the rotation axis normal to P4 was formed with the rotation of 15 degrees counterclockwise to the DM.

The value of stress in (001) LNT specimen was always higher than that in (111) RT specimen. In addition, the mean cell size of (111) RT specimen was larger than that of (001) LNT specimen. So it would be mentioned that the dislocation density in the substructure of (001) LNT specimen was higher than that of (111) RT specimen. However, after the annealing the number of the RGs formed in (001) LNT specimen was actually fewer than that of (111) RT specimen. This is caused
by the reason that the cross slip in (001) LNT specimen can take place easily during heating in the annealing process, although it was suppressed in the deformation at LNT. Therefore, it would be judged that annihilation and rearrangement of the dislocations stored in (001) LNT specimen were more facilitated at the stage of recovery than those in (111) RT specimen. In result, recovery of the deformation substructure in (001) LNT specimen proceeded faster, and then stored dislocations necessary for the nucleation of RGs would be much reduced. Thereby, it was considered that the frequency of the nucleation of RGs in (001) LNT specimen was lower than that in (111) RT specimen.

4. Conclusions

Using aluminum single crystal specimens with tensile axes of (001) and (111), deformation and recrystallization behavior depending on tensile orientation and deformation temperature were investigated with special attention to the occurrence of cross slips. The conclusions obtained in the present study were as follows:

(1) In (001) RT specimen, the cross slips with large steps take place very easily, because all the eight primary slip systems have an appropriate cross slip system geometrically. Therefore the excess dislocations are not so much stored in the deformation substructure.

(2) In (111) RT specimen deformed in tension, cross slips cannot be observed at least macroscopically, because compressive strain is generated by the cross slip from the primary slip system to the cross slip one and vice versa. The occurrence of cross slips is not observed either in (001) LNT specimen because of the temperature dependence. Therefore, the dislocations introduced by the active slip systems in these specimens are tangled with each other with high dislocation density in the cell walls. In result, many dislocations necessary for the nucleation of RGs are stored in the deformation substructures. The former specimen has layered cell structure, while the latter one polygonal or round cell structure.

(3) The easiness or difficulty of recrystallization is closely related to the difficulty or easiness of cross slips during the deformation, which depends on the tensile orientation and the deformation temperature. In particular, the RGs are formed at lower annealing temperature and shorter time when the occurrence of cross slips is suppressed during the deformation.

(4) The formation of RGs in the multiple slipped structure could not be explained by the strain-induced boundary migration theory or the coalescence theory, but the (111) rotation recrystallization mechanism.

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