Recrystallization Textures in Single Crystal of Al-1% Mn Alloy Rolled to (123)[412]

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Rolling and recrystallization textures of Al-1 wt% Mn alloy single crystal with (123)[412] as the initial orientation were investigated after 90% cold rolling and annealing at various temperatures. A homogeneous rolling texture with the principal orientation of (167)[923] was obtained. At 653 K where the competition between recrystallization and precipitation occurs, recrystallized grains whose orientations were determined by micro Laue method have the same orientation as the deformed matrix. The formation of these recrystallized grains are considered to be a result of recrystallization in situ. Annealing at temperatures above 673 K where recrystallization precedes precipitation leads to the generation of a number of orientations as (476)[221], (123)[432], (739)[1219], (437)[704] and (011)[611], all of which are related to the deformed matrix by rotations around $\langle 111 \rangle$ axes. These orientations result from selective growth owing to the existence of Mn and a small amount of Fe in the solid solution state.

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I. Introduction

Recrystallization textures of aluminum sheet are strongly influenced by homogenization, hot rolling and annealing temperature. For instance, recrystallization textures of commercially pure aluminum are in general composed of the cube texture $\{001\}\langle 100 \rangle$ and the retained rolling texture, R-orientation (near $\{123\}\langle 412 \rangle$)⁽¹⁾. The formation of R-orientation⁽²⁾⁽³⁾ is caused by the precipitation of Fe in the solid solution and consequently the generation and growth of cube grains are suppressed. Hornbogen *et al.*⁽⁴⁾ distinguished between discontinuous (conventional) recrystallization and continuous (in situ) recrystallization for the first time. At the temperatures where recrystallization precedes precipitation, discontinuous recrystallization leads to the preferential development of grains of cube and other orientations⁽⁵⁾, both of which are related to the deformed matrix by 40° rotation around $\langle 111 \rangle$ axes. On the other hand, continuous recrystallization gives rise to an annealing texture similar to the rolling texture at the temperatures at which there is con-

siderable competition between recrystallization and precipitation.

Recrystallization in situ was first found out by Crussard⁽⁶⁾. He reported that after annealing, lightly deformed aluminum single crystals and polycrystallites showed some recrystallized grains whose orientations were identical with the initial orientations of the specimens before deformation. He also reported that recrystallization did not suddenly start but occurred without nucleation after recovery took place. Unfortunately, distinct evidence of recrystallization in situ cannot be found in his papers. On the other hand, except the work of Itoh *et al.*⁽³⁾⁽⁵⁾ on an Al-Fe alloy polycrystalline specimen in an attempt to analyse R-orientation, observation of the in situ recrystallized grain has seldom been performed on aluminum alloys heavily deformed and particularly when recrystallization is effected by precipitates during annealing. In general, the formation of the in situ recrystallized grain is hardly distinguishable from that of discontinuously recrystallized grains in polycrystalline material because the 40° $\langle 111 \rangle$ rotational relationship exists between the rolling orientations and furthermore the spread of the rolling texture is wide.

In this paper, in order to investigate the formation of grains with the same orientation as the rolling orientation by recrystallization

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in situ, a (123)[41 $\bar{2}$] Al-1 wt% Mn alloy single crystal was chosen because this orientation is one of the stable orientations in heavily rolled aluminum and its alloys, and the orientational change during rolling is relatively small⁽⁷⁾⁽⁸⁾. There is an additional reason that recrystallization of Al-Mn alloy^{(9)~(11)} is markedly retarded by precipitates. The recrystallization textures obtained at high temperatures were also discussed.

II. Experimental Method

The alloy, made of 99.99% purity aluminum and Al-9.95% Mn master alloy, was melted in a graphite crucible and a single crystal of 40 mm diameter, 160 mm length was grown by the Bridgman method. The chemical analysis of the alloy is shown in Table 1. Homogenization was performed at 913 K for 360 ks, and the crystal was quenched into water. Although Al₆(Mn, Fe) primary inclusions were not perfectly dissolved into the matrix, the alloying elements were kept in solid solution to some extent by the treatment. Specimens of size 30×20×7 mm³ were cut to (123)[41 $\bar{2}$] and rolled to 90% reversely end to end between passes with machine oil as a lubricant. Rolling was performed by a mill with a roll diameter of 150 mm. The reduction per each pass was determined by the ratio $l_c/d=1.6$ (l_c : contact length between roll and specimen, d : specimen thickness before rolling). According to Lücke *et al.*⁽⁸⁾ and Asbeck *et al.*⁽¹²⁾ the above-mentioned rolling process suppresses the formation of inhomogeneous texture resulting from severe friction between roll and specimen. Subsequently, 90% cold rolled specimens were chemically etched up to half of the thickness before annealing. Isothermal recrystallization behavior was examined by means of Vickers hardness measurements and optical and electron microscopic observations. Textures were determined at the central layer of specimens

by a conventional X-ray diffractometer and intensity was normalized by the intensity of the Al-1% Mn powder specimen. In order to determine orientations of recrystallized grains a micro Laue camera equipped with a collimator of a diameter of 100 μ m was used. Laue diffraction patterns were obtained by unfiltered Cu radiation.

III. Results and Discussion

1. Rolling texture of (123)[41 $\bar{2}$] single crystal

The rolling texture of 90% cold rolled (123)[41 $\bar{2}$] Al-1% Mn alloy single crystal is shown in Fig. 1. The principal orientation (167)[92 $\bar{3}$] is identical with that found in the experiment by Lücke *et al.*⁽⁸⁾ on the pure aluminum single crystal with a similar initial orientation. In addition to this orientation, other orientations considered as the "ghost peak" due to the diffraction of white X-rays are also observed. Although primary inclusions still remained before rolling, the identity of the principal orientation implies that they are not likely to be of importance in affecting the formation of the rolling texture. The reduction per pass determined by $l_c/d=1.6$

Fig. 1 {111} pole figure of 90% rolled (123)[41 $\bar{2}$] single crystal.

Table 1 Chemical composition of specimen (wt%).

Mn	Fe	Si	Cu	Al
1.13	0.019	0.014	0.002	bal.

presented a homogeneous texture.

2. Recrystallization behavior of heavily rolled single crystal

Isothermal annealing curves for 90% rolled single crystal are shown in Fig. 2. Optical and electron microscopic observations were performed at various annealing stages to investigate the structural change at 653 K where slow softening is observed. A representative photograph shows that the structure is a mixture of recrystallized grains and well-defined subgrains, and that particles larger than 0.5 μm are distributed in the recrystallized grain (Photo. 1-(a)). Electron diffraction analy-

sis showed that these particles are the equilibrium phase $\text{Al}_6\text{Mn}^{(11)}$. As seen in Photo. 1-(b), large particles scattering in and outside the subgrains grow up to more than 1 μm . Photograph 2, an optical microstructure of the specimen annealed for 356.4 ks indicates the presence of the deformed matrix although the hardness of the specimen is identical with that of the fully recrystallized one. The jagged boundaries implies the pinning effect of particles which are formed during annealing.

On the other hand, at the temperatures above 673 K where recrystallization precedes precipitation specimens fully recrystallized within 0.72 ks and the resulting textures would be controlled by discontinuous recrystallization.

3. Texture due to recrystallization in situ

Figure 3 shows the texture of specimen annealed for 1.21 Ms at 653 K. This temperature, from the above results, is considered as in the temperature range of the competition between recrystallization and precipitation. Such a temperature is consistent with the results obtained by Nagahama *et al.*⁽⁹⁾ and Goel *et al.*⁽¹⁰⁾ on Al-Mn alloy polycrystallites. The rolling orientation (167)[92 $\bar{3}$] is strongly retained and the presence of other orientations is also observed. The microstructure of the specimen at the rolling plane is shown in Photo. 3. Numerous recrystallized grains with jagged boundaries are observed. Since these grains seem to take part in the formation of the remaining rolling texture, their orientations

Fig. 2 Isothermal annealing curves at various temperatures.

Photo. 1 Transmission electron micrographs of specimens annealed at 653 K for (a) 21.6 ks and (b) 172.8 ks showing the structure composed of recrystallized grain and subgrains.

Photo. 2 Microstructure of specimen annealed at 653 K for 356.4 ks showing recrystallized grains with jagged boundaries.

Fig. 3 {111} pole figure of specimen annealed at 653 K for 1.21 Ms showing the strongly remaining rolling texture.

were determined by micro Laue method and the results obtained are shown in Fig. 4. Symbols used in Fig. 4 are the same one as that marked in the recrystallized grains in Photo. 3. It is to be noted that the grains which are marked by the filled circles are situated at (167)[923], and that even after recrystallization the orientation (the principal orientation of the rolling texture) does not change. It was also found that grains with the same orientation as the deformed matrix cannot be distinguished

from others by optical observation because their shapes are almost identical. On the other hand, orientations of the rest of the recrystallized grains belong to (476)[221], (123)[452] and (011)[611] whose relations with the deformed matrix will be discussed later.

The formation of the annealing texture with the same principal orientation as the rolling texture is considered to occur in the following manner. After heavy deformation, annealing at 653 K leads to the precipitation of Al_6Mn particles which, during the early stage of annealing, precipitate on such lattice defects as dislocations and subboundaries. This results in the stabilization of substructures. Consequently, the migration of dislocations and subboundaries is retarded and hence the nucleation for recrystallization should be the very slow and intermittent coarsening⁽¹³⁾⁽¹⁴⁾ of subgrains by coalescence and sub-boundary migration, which take place simultaneously with the growth of precipitates. In this mechanism, a very long annealing time is needed before the specimen becomes recrystallized and finally the orientational change does not occur since no high angle boundary is formed. The above-mentioned consideration is supported by the transmission electron micrographs shown in Photo. 1, the jagged boundaries of recrystallized grains in Photos. 2 and 3, and by the hardness measurement in Fig. 2. All of these results indicate apparently the remarkable pinning effect due to the fine precipitates. In the present work, however, it was found that the nucleation for recrystallization is not completely inhibited since there are the remaining primary inclusions which could work as nucleation sites. A number of fine recrystallized grains are found in the neighborhood of the primary inclusions (Photo. 3). From the results of the X-ray pole figures (Figs. 3 and 4) these grains would be such orientations as (739)[1219], (123)[452], (011)[611] but probably not the same orientation as the deformed matrix. Therefore, not only the remaining rolling texture but also other orientations exist in the pole figures shown in Figs. 3 and 4. Considering from the intensity of these orientations, it is noticeable that (167)[923] orientation overcomes others by its maximum intensity of 70.

Photo. 3 Microstructure at rolling plane of specimen used in Fig. 3. Grains with filled circle have the same orientation as the deformed matrix.

Fig. 4 The $\{111\}$ pole figure determined by a micro Laue method of grains marked by circles in Photo. 3.

The difference in intensity could be explained by the fact that the well-pronounced pinning effect of finely stable Al_6Mn precipitates inhibits the nucleation and/or growth of grains by discontinuous recrystallization, and allows recrystallization to occur mainly by continuous one. Itoh *et al.*⁽⁵⁾ observed in an experiment on Al-Fe alloy that the higher the stability of the precipitate, or the stability of the substructure, the more easily the formation of grains with the same orientation as the deformed matrix occurs. In the present study, the nucleation and growth by discontinuous recrystallization which is controlled by the $40^\circ\langle 111 \rangle$ rotational relationships would markedly be suppressed, and hence a recrystallization texture similar to the deformation texture is permitted to be formed easily.

As seen in Photo. 3, almost none of recrystallized grain is found in the regions with densely distributed particles. Although the X-ray diffraction photographs obtained from these regions presented Debye rings due to $\text{CuK}\alpha$ radiation, hardness of the specimen is the same one of the fully recrystallized specimen (Fig. 2) and an electron microscopic observation showed that these regions consist of subgrains

larger than $5\text{ }\mu\text{m}$ with a lower density of dislocations in the grain interior. These regions, therefore, are considered to be on the extension of recovery. Such a structure should be the characteristic of recrystallization in situ and takes a great part in formation of the remaining rolling texture.

4. Textures due to discontinuous recrystallization

Very similar textures of specimens annealed at 673 and 873 K for 720 and 5 s, respectively, are shown in Fig. 5. At these temperatures, since recrystallization completes within a short time (Fig. 2), the effect of precipitates is considered to be negligible (strictly speaking, annealing at 673 K leads to the formation of fine particles on high angle boundaries but the effect of them is believed to be small or negligible because precipitation occurs after remarkable growth of recrystallized grains). The rolling texture, therefore, is not retained. The recrystallization textures mainly consist of (476)[$\bar{2}2\bar{1}$], (739)[$12\bar{1}9$] and (011)[$6\bar{1}1$] orientations. The relationships between these components and the deformed matrix were found by calculation from 24 sets⁽¹⁵⁾ of rotational axes. Of 24 axes calculated for one orientation only axis most close to $\{111\}$ poles of the deformed matrix was selected and then plotted in Fig. 6. All of the selected axes are within 10° from the four poles of $\{111\}$ presenting the deformed matrix. For instance, the rotational axis of (476)[$\bar{2}2\bar{1}$] component lies to [$\bar{1}\bar{1}1$] within 4° and hence it could be considered to be [$\bar{1}\bar{1}1$] (the calculated axis is approximately $[4\bar{4}5]$). Table 2 shows the rotational relationships of five orientations in the recrystallization textures of Fig. 5 with respect to the deformed matrix. Except (011)[$6\bar{1}1$] orientation, the remainder has the common about $40^\circ\langle 111 \rangle$ rotational relationships. Rixen *et al.*⁽¹⁶⁾ reported, in an experiment on Al-Mn alloy polycrystalline specimens, that recrystallization textures are related to the deformed matrix by the above-mentioned relationships. The (011)[$6\bar{1}1$] orientation can be obtained by a rotation of about 152° around an axis of $[564]$ (it could be approximated as $[111]$). Of the four $\langle 111 \rangle$ axes, only rotation

Fig. 5 {111} pole figures of specimens annealed at 673 and 873 K for 720 s and 5 s, respectively. They show the presence of recrystallization orientations involving the $40^\circ \langle 111 \rangle$ relationship.

(a) 673 K, 720 s, (b) 873 K, 5 s

around $[1\bar{1}1]$ is not found. This can be explained by the fact that there is the effect of the anisotropy of boundary mobility⁽⁷⁾ which may be related to the diffusion rate of Mn and a small amount of Fe solutes atoms along the

Fig. 6 {111} pole figure showing positions of calculated rotational axes for various orientations. ①, ⊖, ⊙, ⊗ and ● are rotational axes for (476) $\bar{2}\bar{2}\bar{1}$, (123)[452], (437)[704], (739)[12 $\bar{1}$ 9] and (011)[6 $\bar{1}$ 1], respectively.

Table 2 Relation between recrystallization orientations and the deformed matrix.

Orientation	Angle*	Axis	Direction
(476)[22 $\bar{1}$]	40.0°	[1 $\bar{1}$ 1]	anti-clockwise
(123)[4 $\bar{5}$ 2]	43.2°	[1 $\bar{1}$ 1]	clockwise
(437)[704]	42.2°	[$\bar{1}$ 11]	clockwise
(739)[12 $\bar{1}$ 9]	43.6°	[$\bar{1}$ 11]	anti-clockwise
(011)[6 $\bar{1}$ 1]	151.7°	[111]	anti-clockwise

(* calculated value)

boundary.

On the other hand, it is to be noted that the intensity of the (476)[$\bar{2}2\bar{1}$] orientation which was also observed in pure aluminum single crystal with the same initial orientation increases with increasing temperature. This implies that (476)[$\bar{2}2\bar{1}$] may be the preferred orientation of the recrystallization texture in the (123)[$41\bar{2}$] Al-Mn single crystal heavily rolled and then annealed at high temperature. A reverse result, however, is obtained in the (011)[$6\bar{1}1$] orientation whose intensity decreases from 11 to 1 when annealing temperature is increased from 673 to 873 K. The annealing texture at 773 K for 15 s, which was

not shown here, also presented an intermediate intensity between the two. In general, the difference of recrystallization textures due to annealing temperatures are explained by the fact that the precipitation behavior and the drag effect of alloying elements vary depending on annealing temperature. The experiments performed at 673 and 873 K are connected with the recrystallization textures which are considered to be unaffected by precipitates. A possible account, therefore, for the change in intensity of recrystallization orientations is the possibility of the effect of Mn and Fe solute atoms on grain boundary migration. It appears that another explanation may be the temperature-dependence of the rotational relationship which could cause the alteration of the texture. However, considering the unchanged orientations in the textures of Fig. 5 and the fact that the rotational relationship in heavily rolled Al-Mn⁽¹⁶⁾ and Al-Fe⁽³⁾⁽⁵⁾ alloy polycrystallites does not alter with respect to annealing temperature, such a dependency should be excluded.

As shown in Fig. 5, a number of recrystallization orientations exist in Al-Mn alloy while one component of (476)[$\bar{2}2\bar{1}$] (Lücke *et al.*⁽⁸⁾ stated it as (467)[$\bar{2}1\bar{2}$]) and randomly distributed orientations were observed by Lücke *et al.* on the pure aluminum single crystal. The presence of Mn and a small amount of Fe in solid solution should play an important role in formation of the recrystallization texture, because it is well known that in aluminum with high concentrations of impurities and alloying solutes, no random recrystallization texture is allowed to develop on annealing after heavy deformation. Solute Mn and especially Fe seem to have an effect on suppressing randomly oriented grains which nucleate and grow easily in high purity aluminum. In the present work, however, it is necessary to note that the textures in Fig. 5 should be attributed to selective growth.

IV. Summary

(1) The rolling texture of Al-1% Mn alloy single crystal with the initial orientation

(123)[$\bar{4}1\bar{2}$] is of a homogeneous texture and has (167)[$\bar{9}2\bar{3}$] as the principal orientation.

(2) Annealing at 653 K where the competition between recrystallization and precipitation occurs, gives rise to the formation of grains with the same orientation as the deformed matrix. The presence of such grains are resulted from recrystallization in situ due to the strong pinning effect of the stable Al₆Mn precipitates.

(3) On annealing at temperatures above 673 K, a number of recrystallization orientations related to the deformed matrix by rotations around $\langle 111 \rangle$ are obtained. Particularly, one of these orientations, (476)[$\bar{2}2\bar{1}$] is considered to be the principal preferred orientation in heavily deformed and recrystallized Al-Mn alloy (123)[$\bar{4}1\bar{2}$] single crystal. The orientations obtained at high temperatures should be regarded as a result of selective growth.

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