Electron Back-Scatter Pattern Analyses of a Recrystallized Al-4mass%Mg Alloy Sheet†

Toshiya SHIBAYANAGI* and Masaaki NAKA**

Abstract

Spatial distributions of grain orientation and grain boundary character distribution in a recrystallized Al-4mass%Mg alloy sheet have been investigated using the Electron Back Scatter Pattern (EBSP) method. The evaluation of microstructure was performed for the specimen recrystallized at 773K for 1ks. Weak cube textures were observed for the specimen taken from the central portion of the thickness, and sheared texture component tended to be dominant in the surface region of the sheet. Cube oriented grains existed in both isolated and aggregated sates. The cluster of cube grains tended to contain low angle grain boundaries. Observation of grain boundary migration on the same area revealed heterogeneous behavior of triple junctions resulting in a wide variety of microstructural change.

KEY WORDS: (EBSP)(Al-4Mg alloy)(recrystallization)(texture)(grain boundary character distribution) (grain boundary migration)(triple junction)

1. Introduction

Microstructure of polycrystalline materials is well characterized with (1) kinds of phases, (2) shape and size of grains, (3) crystallographic orientation of grains, and (4) structure and/or character of interfaces. These microstructural parameters strongly affect mechanical properties of materials. Therefore much attention has been focused on the control of microstructure in order to produce strong materials.

These microstructural parameters mainly represent "macroscopic" aspects of polycrystalline structure, in other words, statistical parameters such as the average grain size, pole figures for texture and grain boundary character distribution.

Recently a new concept of spatial distribution of microstructural parameters such as "microscopic" and "mesoscopic" textures has been proposed[1]. Each parameter represents the spatial distribution of grain orientation and grain boundary characters, respectively. These parameters are useful for describing local microstructures that show different aspects from each other in every region of the specimen.

Complicated changes of each local microstructural parameter as well as the statistical parameters are known to take place and affect each other in the evolution of microstructure at elevated temperature. This phenomenon is critical in the case of normal or abnormal grain growth during annealing of cold rolled metals, heat affected zone and weld metals of joints.

In order to design the most suitable microstructure for given materials or joints it is quite important to elucidate the role of these microstructural parameters in the evolution of microstructrue like grain growth, while little information has been reported so far.

Recently the Electron Back-Scatter Pattern (EBSP) technique has been developed and introduced for the identification of the spatial distribution of microstructural parameters[2, 3].

The present paper attempts to reveal the characteristics of the local microstructural parameters and especially to find trigger conditions in the grain growth of the recrystallized Al-4mass%Mg alloy.

2. Experimental procedure

A sheet of cold rolled Al-4mass%Mg alloy was prepared by Sumitomo Light Metals Co.Ltd. The reduction was 90% in thickness. Specimens for heat treatment, 5 mm in width and 7mm in length, were machined from the cold rolled strip and then sealed in quartz tubes evacuated to less than 1mPa. The recrystallization treatment was carried out at 773K for 1ks, yielded an equi-axed polycrystalline structure with an

† Received on July 1, 2001
* Associate Professor
** Professor

Transactions of JWRI is published by Joining and Welding Research Institute of Osaka University, Ibaraki, Osaka 567-0047, Japan.

average grain size of 100 μm.

Evaluation of microstructure for the recrystallized specimen was performed by means of optical microscopy and the EBSP method focusing on the spatial distributions of grain orientation and grain boundary characters. Two regions of the annealed specimens were observed in the present work; the surface and the center of the thickness. Each annealed specimen was mechanically polished to the determined thickness and then electrochemically polished at –20 ℃ using an ethanol solution containing $HClO_4$ at 10 volume %.

The EBSPs were taken in a SEM (Jeol, JSM-6400) with an accelerating voltage of 30kV, and each pattern was analyzed with a specialized software (OIM™ version 2.2) provided by TSL Co. Ltd.

The behavior of grain boundaries and triple junctions was observed on the same area of the each specimen analyzed. The additional heat treatment was performed in vacuum for 100 second at the same temperature as that of recrystallisation. After this heat treatment, the surface of the specimens were observed by SEM to investigate the displacement of grain boundaries and triple junctions.

3. Results and discussion

3.1 Orientation distribution in the center region of thickness

Distribution of grain orientation was firstly analyzed based on the EBSP data taken from the specimen recrystallized at 773K for 1ks, and a typical result is shown in **Fig.1**

Figure 1 (a) shows discrete pole figures for 111, 100 and 110 components. Each point corresponds to the EBSP data, i.e. the measured points. There is a weak cube texture in this specimen, which is consistent with other reports on Al-Mg alloys. The data highlighted with green color are close to the cube orientation of (100)<001> component. The deviation angle from the just cube orientation of each green point is less than 15 degrees.

Figure 1(b) highlights the spatial distribution of cube oriented grains with green color corresponding to the plot in Fig.1(a). Each grain or each measured point has different gradations of green color, darker green representing the orientation is closer to the just cube orientation. Taking note of the existence of cube oriented grains, there are isolated grains and particularly aggregations of grains with the similar orientation component, which will be called "clusters" in the present paper.

Grain boundaries with red and black color correspond to low angle and high angle boundaries, respectively. The isolated cube grains tended to be surrounded by high angle boundaries, and low angle boundaries are abundant in the cube clusters.

3.2 Grain boundary character distribution in the center region of thickness

Grain boundary character distribution was then analyzed and the result is shown in **Fig.2.** Figure 2(a) shows a result of statistical analysis. The sigma value is calculated based on the coincidence boundary theory[4]. The figure indicates that the fraction of boundaries tended to decrease as the sigma number increase.

Fig.2(b) represents a spatial distribution of coincidence and random boundaries. Each number beside a boundary denotes the sigma value, and "L" means low angle boundaries with the misorientation angle less than 15 degrees. In the present work boundaries with the sigma value above 23 are included into random boundary. Coincidence boundaries distribute heterogeneously, for example, the $\Sigma 3$ boundaries tends to aggregate.

Triple junctions composed of three boundaries also show wide variety. Each junction has different dihedral angles between neighboring boundaries, implying that some junctions are not in the equilibrium state viewed from the energy balance of boundaries at the junction. But this analysis cannot tell active from passive state, and this will be analyzed and discussed in the later sections of the present paper.

3.3 Orientation distribution in the surface region

As the cold rolling process brings about a complicated strain distribution throughout the thickness of the strip, the orientation distribution should be understood as a function of the position in the thickness. The surface region was then analyzed and a typical result is shown in **Fig.3.**

Discrete pole figures shown in Fig.3(a) represent the weak texture of ND rotated-cube component, i.e. a rotation of cube orientation by about 45 degrees around the normal direction of the surface. The data with ND rotated-cube orientation is highlighted with yellow color. This texture component has been often observed for recrystallized sheets after severe cold rolling[5].

The corresponding spatial distribution of grain orientation is represented in Fig.3(b). The ND rotated-cube grains with yellow color exist with three cube-oriented grains with green color. There are isolated grains as well as the clusters. Low angle boundaries with red color tended to exist in the clusters.

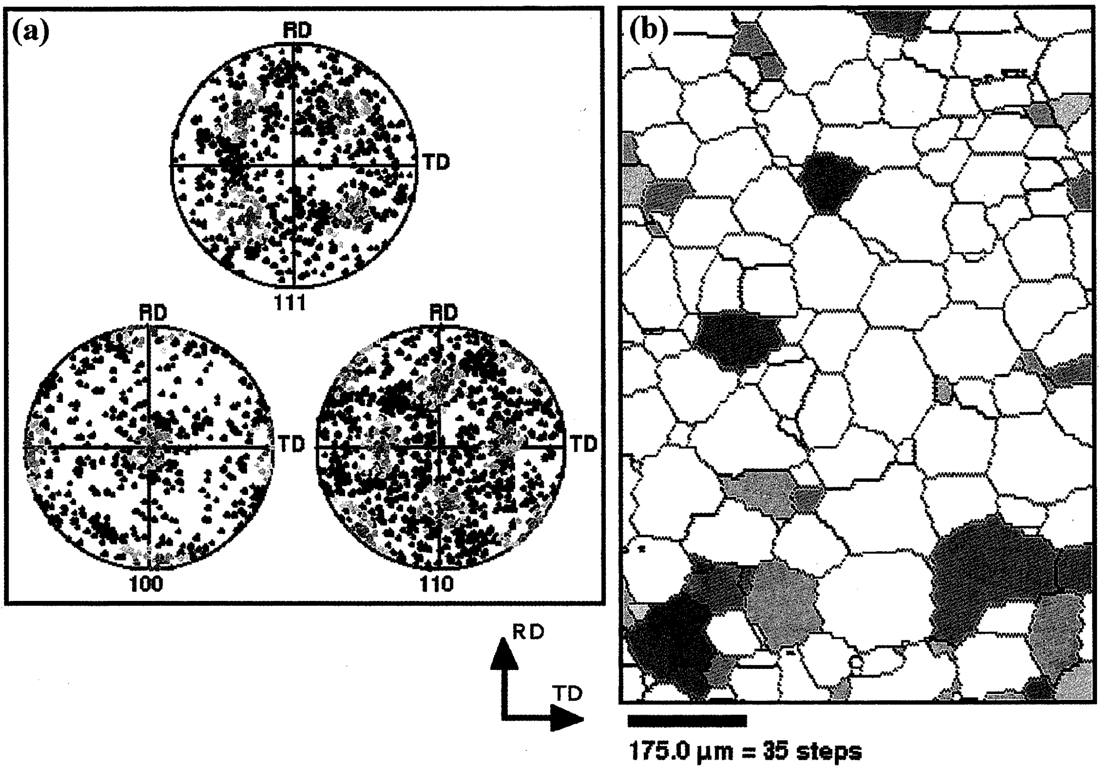


Fig.1 Texture analysis (a) and the spatial distribution (b) of grain orientation in the center region of the thickness of the Al-4mass%Mg alloy sheet recrystallized at 773K for 1ks.

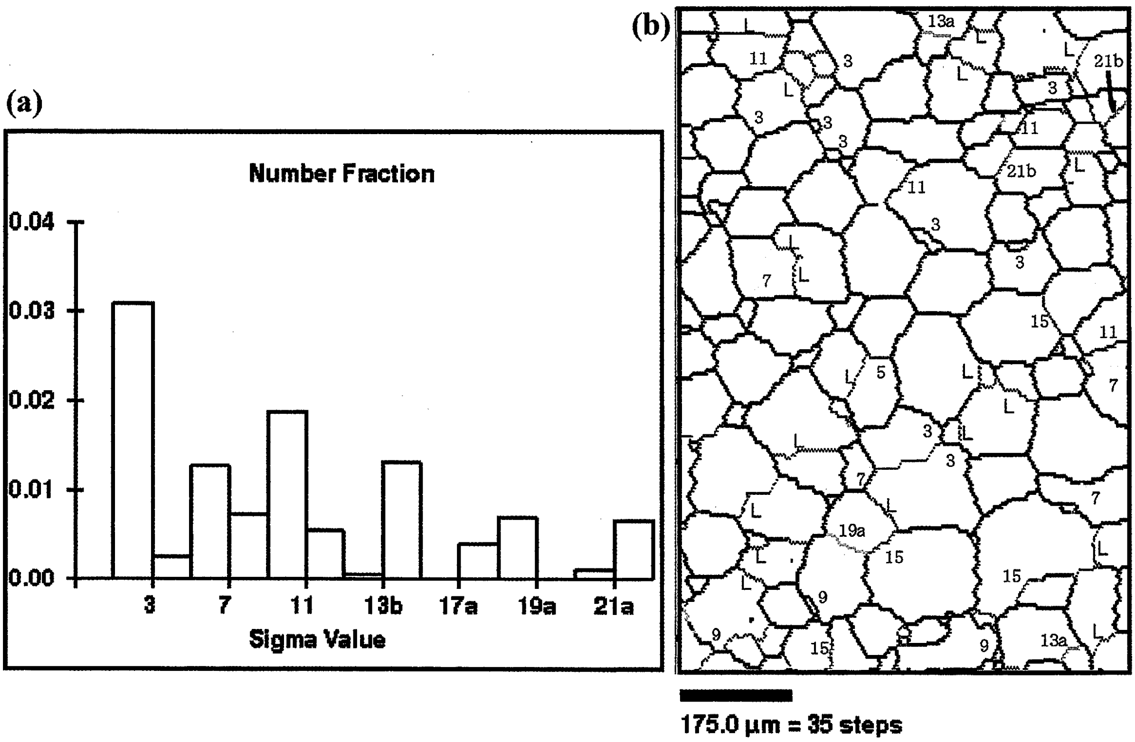


Fig.2 Grain boundary character distribution in the center region of the thickness of the Al-4mass%Mg alloy sheet recrystallized at 773K for 1ks. (a): fraction of low angle boundaries and the coincidence boundaries with the sigma value less than 21, (b):spatial distribution of grain boundary characters.

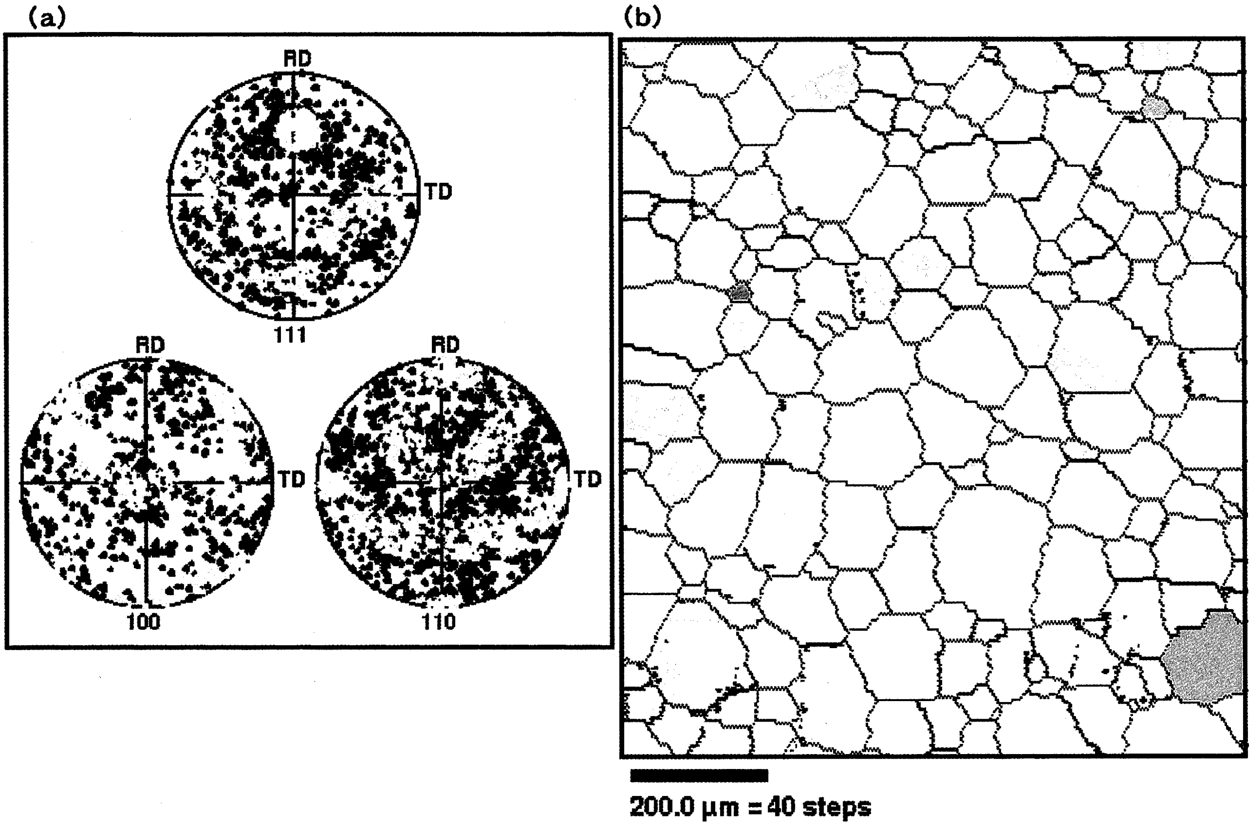


Fig. 3 Texture analysis (a) and the spatial distribution (b) of grain orientation in the surface region of Al-4mass%Mg alloy sheet recrystallized at 773K for 1ks.

These characteristics of the spatial distribution seem to be similar to those observed for the central position of the thickness. A drastic change of texture component has been observed in pure Al thin foil[6], but the present study did not reveal any transient region from the center to the surface of the specimen.

3.4 Migration behavior of grain boundaries and triple junctions

The spatial distributions explained so far represent just a static status of microstructure, not dynamic changes to be occurring afterwards as the annealing of the specimen proceeds. Since there are many kinds of triple junctions, there would be heterogeneous evolution of microstructure and too many remaining unclear key factors or trigger factors for a drastic change of microstructure to show up.

The additional annealing at the same temperature was performed for the same specimen analyzed in Figs. 1 to 3. The annealing time was 100s. Back-scattered electron images (BEIs) of the surface of the additionally annealed specimen is shown in **Fig.4**. In this figure, both the original position and the present position of grain boundaries and triple junctions are visible. Since the slight etching of the specimen after the recrystallization treatment made a shallow groove of each boundary, the grooved line represents the former position. The present position of boundaries is recognized by the difference of darkness in the BEI, since the contrast of the BEI is sensitive to the grain orientation. Many boundaries and triple junctions are moving, but not all. Some typical cases will be explained as follows.

Figure 5 shows a passive triple junction pointed with an arrow, where no migration of boundaries is taking place. As indicated in Fig.5(b) that represents a part of the result of EBSP analysis explained in Fig.1(b), every boundary around this junction is a random boundary. This result suggests that random boundaries with high energy and high mobility do not always migrate preferentially.

Figures 6 and 7 shows an active triple junction pointed with an arrow, where grain boundary migration is proceedings. An isolated cube-oriented grain with green color shown in Fig.6 (a) is now shrinking. The grain is surrounded by random boundaries that are mobile in the local condition of microstructure. Three red grain boundaries are also shown to be connecting to form a

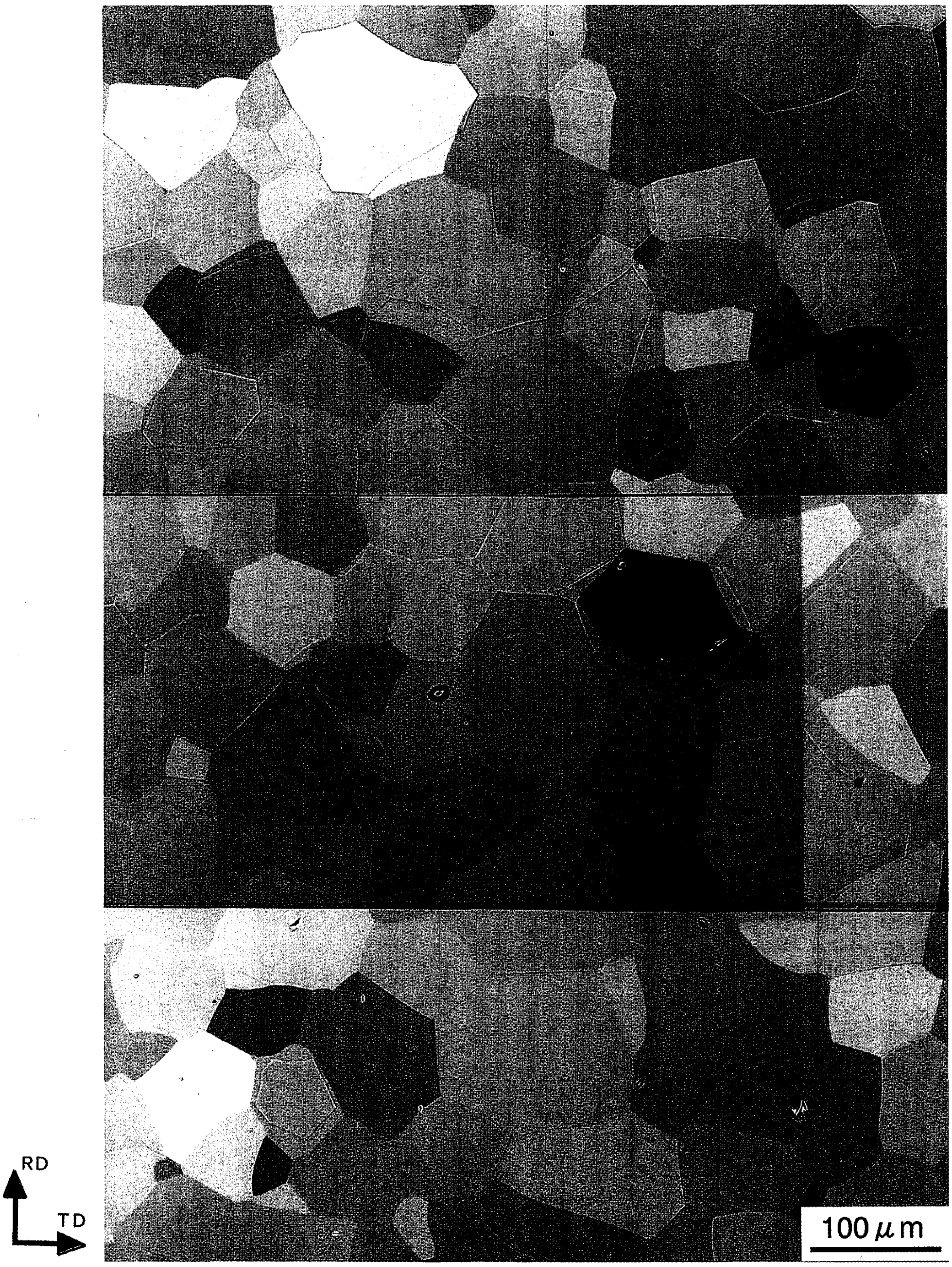
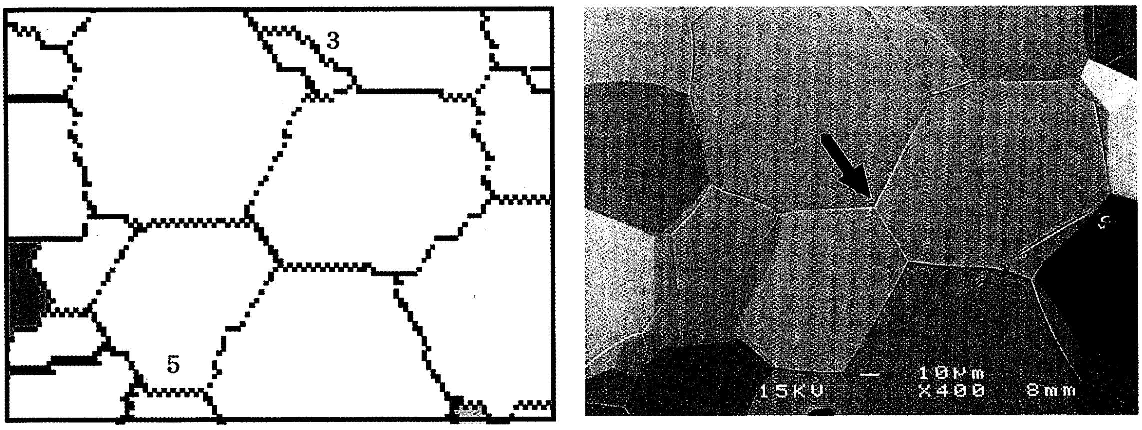
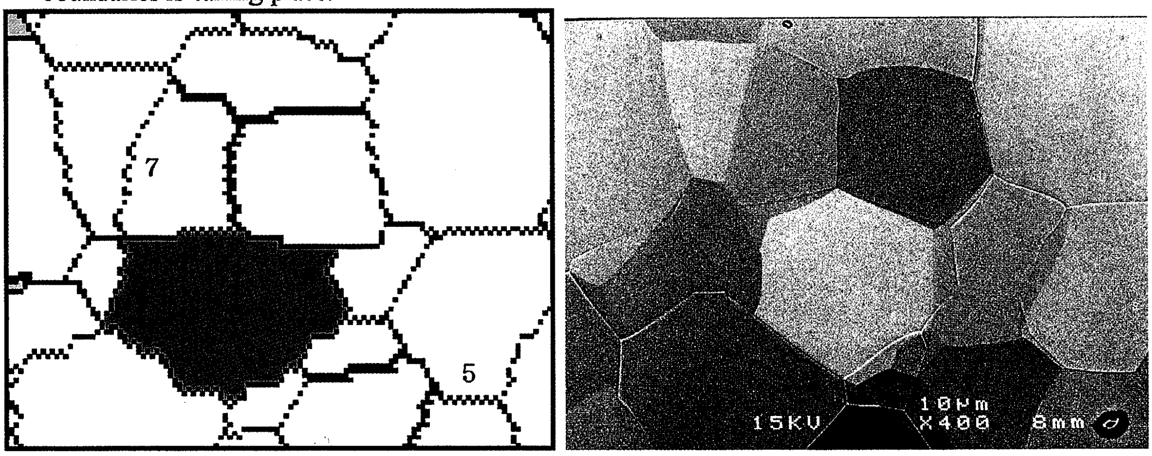


Fig. 4 Back-scattered electron image of the surface of the additionally annealed specimen, showing the migration of grain boundaries and triple junctions.



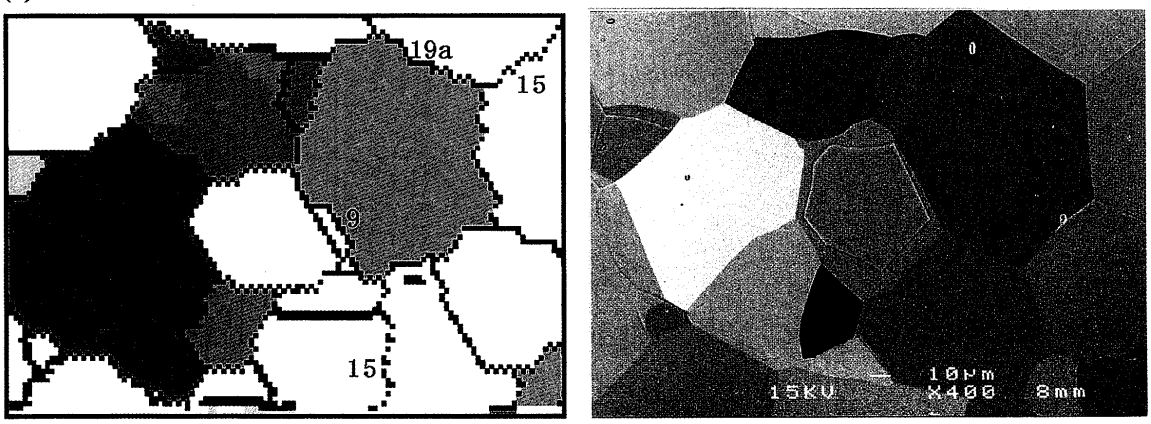
(a) (b)

Fig.5 An example of passive triple junction observed for the specimen additionally annealed.
(a): spatial distribution of grain boundary character of the specimen before the additional annealing.
(b): BEI of the same area after the heat treatment. Arrow shows a passive junction, where no migration of boundaries is taking place.



(a) (b)

Fig. 6 An example of the active triple junction around an isolated cube-oriented grain.
(a):spatial distribution of grain boundary character of the specimen before the additional annealing.
(b): BEI of the same area after the heat treatment.



(a) (b)

Fig. 7 An example of the active triple junction around a cluster of cube-oriented grains.
(a):spatial distribution of grain boundary character of the specimen before the additional annealing.
(b): BEI of the same area after the heat treatment.

triple junction that is not moving. This is another case of passive triple junction composed of the similar grain boundary characters. Note that the curvature of each grain boundary is changing near junctions, suggesting the local accommodation of energy balance of boundaries.

Figure 7 shows a cluster of cube-oriented grains that is also being invaded by a contacting non-cube grain surrounded by random boundaries. This is quite an important result, suggesting that all the clusters cannot grow preferentially. Some other microstructural factors should be taken into account for the determination of the critical growth condition of grains.

4. Discussion

4.1 Cluster of cube orientation

As shown in Fig.1 and 3, the volume fraction of cube oriented grains in the center part of thickness was more than in the surface region. The difference of orientation distribution is attributed to the strain distribution throughout the thickness of the cold rolled strip. The surface region contains shear components resulting from the severe constraint caused by the friction with the roll during cold working.

The micro-texture can tell us the other specific aspect of the microstructure shown in Fig.3. As explained previously, one of the typical characteristics observed for the present specimen is the cluster of grains with the cube or the other orientation components. The cube clusters should be strongly related to the formation mechanism of texture[7,8], which extensive work has been trying to elucidate based on the concept of macro- and micro-texture. The EBSP analyses are quite effective for understanding the spatial distribution of each microstructural parameter. However the roles of each spatial distribution in the evolution of microstructure need much more work.

The role of cluster has been reported for recrystallized Al-0.3mass%Mg alloy by K.Matsumoto et al, explaining that the clustering is an effective microstructural condition that should enhance the preferential grain growth[9]. They have claimed two major roles for the clustering: the size advantage effect and the alternation of local grain boundary character distribution around triple junctions. The latter effect will be discussed in the following section.

4.2 Role of triple junctions: a trigger effect

The essential and elemental process of grain growth is grain boundary migration that is known as a function of grain boundary structure. Generally grain boundary migration is described using the concept of driving force and mobility[10]. The mobility of a grain boundary is well known to be affected by grain boundary structure, for example coincidence boundaries can move faster than other boundaries such as low angle boundaries or random boundary[11-13]. But this phenomenon is dominant in the limited condition of the amount of solute atoms segregating at the interface.

The driving force is rather complicated since the grain boundary structure does not always directly affects this. For example, as seen in Fig. 5, no migration of triple junctions nor boundaries occurred during the additional annealing for the triple junction composed of three random boundaries having almost the same dihedral angle. In this case the energy balance of boundaries at the junction is thought to be in an equilibrium state since the three boundaries possess similar energy level[14]. Therefore this result suggests that there should be quite low driving forces acting on each boundary if the triple junction is stable or in the equilibrium state regardless of the type of boundaries connecting to the junction.

The present work revealed the heterogeneous behavior of triple junctions and grain boundaries. Each triple junction has a different energy balance depending on the types of boundaries, and unstable junctions should have higher driving force than stable ones. Then the second factor comes to be included, that is the mobility of each boundary connecting at the unstable junction. Grain boundary migration can occur only for the boundaries having high mobility unless the driving force is small. Therefore, active triple junctions observed in the present work should satisfy concurrently the following conditions as; (1) to be unstable because of the imbalance of grain boundary energy at junction, and (2) to be composed of grain boundaries with higher mobility.

These active junction should cause a preferential migration and grain growth, and the spatial distribution of the active triple junction must be analyzed for the perfect understanding of the variation of grain growth process or for the prediction of the future state of the given microstructure.

There must be many other unknown factors affecting the migration behavior and grain growth process. The EBSP analysis will help us to find out the trigger parameters that dominate the evolution of microstructure at elevated temperature.

5. Conclusions

Microstructure of a recrystallized Al-4mass%Mg alloy sheet was analyzed by using the EBSP method, focusing on the spatial distributions of grain orientation and grain boundary characters. The following results were obtained.

(1) Weak cube textures were observed for the center part area of the thickness, and sheared texture components tended to be dominant in the surface region of the sheet.

(2) Cube oriented grains existed in both isolated and aggregated status. The cluster of cube grains tended to contain low angle grain boundaries. Triple junctions in the specimens from both regions were revealed to show wide variation depending on the component of boundaries and angles between two neighboring boundaries.

(3) Grain boundary migration showed heterogeneous behavior depending on the status of triple junctions. Random or coincidence boundaries did not always move faster than the others. The active triple junctions were concluded to be a decisive factor for the migration of grain boundaries and the condition for the active junction was to be in unstable status of energy balance and simultaneously be composed of mobile boundaries around it.

(4) The active triple junctions showed heterogeneous spatial distribution in both areas of the thickness of the present specimen.

Acknowledgement

The present work was partly supported by a Grant-in-Aid of the Ministry of Education, Culture, Sports, Science and Technology (Project No. 13450295) . This work is also the results of "Development of Highly Efficient and Reliable Welding Technology" which is supported by the New Energy and Industrial Technology Development Organization (NEDO) through the Japan Space Utilization Promotion Center(JSUP) in the program of Ministry of Economy, Trade and Industry (METI).

REFERENCES

1)V.Randle: "*Microstructure Determination and Its application*", IOP, (1995)
2)D.J.Dingley and V.Randle : J. Mater. Sci., 27(1992)4545.
3)B.L.Adams, S.I.Wright and K.Kunze : Metall. Trans. A, 24A(1993), 819.
4)D.A.Smith: "*Grain Boundary Structure and Properties*", G.A.Chadwick ed., Academic Press, New York, (1976)
5)S.Saimoto et al.: Proc. Textures and Microstructures, 21(1993), 109.
6)F.Seki:J.Japan Inst. Metals, 64(2000),218.
7)S.Kohara, M.N.Parthasarathi and P.A.Beck : Trans. Metall. Soc. A.I.M.E., 211(1958), 875.
8)F.Seki and T.Kamijo : J.Inst. Light Metals, 48(1998),507.
9)K.Matsumoto, T.Shibayanagi and Y.Umakoshi: Acta Mater.45(1997)439.
10)G.Gottstein and L.S.Shvindlerman, "*Grain Boundary Migration in Metals*",CRC Press,(1999)125
11)K.T.Aust and J.W.Rutter: Trans. Met. Soc. AIME, 125(1959),820.
12)H.Nakashima et al.: Tetsu-to-Hagane 82(1996)238.
13)T.Shibayanagi, K.Ichimiya and Y.Umakoshi : Science and Techonlogy of Advanced Materials,1(2000),87.
14)C.Herring: "*Physics of Powder Metallurgy*" , W.Kingston ed. McGraw-Hill, New York(1951), 143.