Effects of Flow Stress and Grain Size on the Evolution of Grain Boundary Microstructure in Superplastic 5083 Aluminum Alloy

Tomotake Hirata*,1, Toshihiro Osa*,2, Hiroyuki Hosokawa*3 and Kenji Higashi

Department of Metallurgy and Materials Science, Osaka Prefecture University, Sakai 599-8531, Japan

The effects of flow stress and grain size on the evolution of grain boundary character distribution (GBCD) at elevated temperatures were investigated using a superplastic aluminum 5083 alloy. In the superplastic region, the percentage of random boundaries was high and was almost unchanged during deformation. In addition, stress and grain-size dependencies on the variation of GBCD were not observed during superplastic deformation. On the other hand, in the dislocation creep region, the percentage of low-angle boundaries gradually increased with increasing strain. Stress and grain-size dependencies on the variation of GBCD were observed and the degree of increase in low-angle boundaries increased with increases in both stress and grain size. This microstructural change could be considered to be influenced by grain boundary sliding (GBS). The degree of increase in low-angle boundaries increased with a decrease in the contribution of GBS.

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1. Introduction

Recently, aluminum has been considered in advanced applications as an energy-saving structural material, because the use of aluminum reduces weight. One such application would be its use in automobiles, which would facilitate transportation; numerous similar examples could be cited in support of employing aluminum as a structural material. However, high-strength aluminum alloys typically exhibit poor ductility at room temperature. An improvement in ductility would be necessary in order to increase the number of potential applications. Such an improvement in ductility can be achieved under deformation conditions at elevated temperatures. The strain-rate sensitivity coefficient of the flow stress, m (=\(\log \sigma/\log \dot{\varepsilon}\)), is a very important factor for determination of the appropriate deformation mechanism. The value of m for superplasticity is high (\(~0.5\)) when grain boundary sliding (GBS) is the predominant mode of deformation.1,2) On the other hand, the value of m for dislocation creep is low when slip is the predominant mode of deformation.3,4) It has been reported that the GBCD is an important factor for the determination of the deformation mechanism; the mechanical properties were shown to be influenced by the GBCD.5–8) Therefore, it is very important to investigate the GBCD of the material in order to improve structural designs. The deformation behavior is dependent on microstructure and test conditions. McNelley et al.9) have reported the stress dependence of changes in misorientation distribution under various test conditions during superplastic and dislocation creep deformations. However, there are no reports addressing the stress dependence of the changes in the GBCD. In addition, grain size is a very important factor at elevated temperatures; however, the grain-size dependence of changes in the GBCD during superplastic and dislocation creep deformations have not been discussed in the literature. Thus, in the present study, materials with different grain sizes were prepared, and the influence of flow stress as well as that of the grain size on the GBCD during both superplastic and slip creep deformation was investigated.

2. Experimental Procedure

The present study made use of 5083 aluminum alloy processed by ingot metallurgy (I/M5083: Al–4.6Mg–0.8Mn–0.1Cr) with different grain sizes. The average grain sizes in I/M5083 were 10, 30, and 60 \(\mu m\). These three materials were received in a sheet form prepared by different thermomechanical treatments, and the sheets were machined into flat tensile samples parallel to the rolling direction. Tensile tests were carried out on an Instron-type testing machine controlled by a computer. These tests were carried out with a range of temperature from 673 to 803 K and a range of strain rate from \(10^{-3}\) to \(10^{-4}\) s\(^{-1}\). Grain boundary character (GBC) was examined by Orientation Imaging Microscopy (OIM), a completely automated system for orientation measurements based on the automated indexing of electron backscatter Kikuchi diffraction patterns.10)

The GBCD for 5083 alloys with different grain sizes before deformation is shown in Fig. 1. In the present study, GBC was divided into three categories, low-angle boundaries (\(\theta < 15^\circ\)), high-angle boundaries, which satisfied the criteria for nearness to coincident site lattice (CSL) relations of up to \(\Sigma 29\) boundaries (\(\theta \geq 15^\circ\)), and random boundaries (\(\theta \geq 15^\circ\)). All materials were composed of a high fraction of random boundaries. The GBCD was almost identical among the three materials.

The contribution of GBS to macroscopic elongation was also estimated quantitatively. Before deformation, the specimen surface was electrolytically polished and marker lines were scribed for sliding measurements on the surface parallel to the tensile axis. The surface appearance of the deformed specimen was inspected by scanning electron microscopy.
(SEM) in order to evaluate the contribution of GBS to total elongation. After deformation, the GBS was estimated experimentally from the offsets of the marker lines or from displacement between adjacent grains perpendicular to the specimen surface, and then the average value was calculated. The contribution of GBS to total elongation, $\varepsilon_{\text{GBS}}$, was calculated by $\varepsilon_{\text{GBS}}/\varepsilon_{\text{TOT}}$, where $\varepsilon_{\text{GBS}}$ was the elongation by GBS and $\varepsilon_{\text{TOT}}$ was the total elongation.

3. Results and Discussion

3.1 Characterization of deformation behavior

From the experimental investigations and theoretical considerations of the steady-state deformation in cases of high temperature creep, the deformation behavior of a material at a variety of high temperatures can be represented by the following constitutive equation, whereby the strain rate depends upon stress, temperature, and grain size:

$$\dot{\varepsilon} = \frac{ADGb}{kT} \left( \frac{\sigma}{G} \right)^n \left( \frac{b}{d} \right)^p,$$

where $\dot{\varepsilon}$ is the steady-state strain rate, $D$ is the appropriate diffusion coefficient ($D = D_0 \exp(-Q/RT)$, where $D_0$ is the frequency factor), $Q$ is the activation energy for the diffusion process, $R$ is the gas constant, $T$ is the absolute temperature, $G$ is the shear modulus, $b$ is the Burgers vector, $d$ is the grain size, $\sigma$ is the applied stress, $p$ is the grain size exponent, $n$ is the stress exponent ($n=1/m$: strain rate sensitivity exponent), and $A$ is the material constant. In the eq. (1), $n$ is a very important factor because it can determine the deformation mechanism at elevated temperatures. It has been reported that in dislocation creep deformation, $D = D_1$, and $p = 0$ in eq. (1)\(^3\)\(^4\) and the stress exponent, $n$, typically displayed a value of about 5 or 3, depending on whether climb of dislocation or viscous glide controlled dislocation was rate controlling in fcc metals and alloys.\(^3\)\(^4\) It has been reported that a decrease in the strain rate sensitivity exponent with a decrease in strain rate results from the presence of a threshold stress, and is not due to the deformation mechanism changes in the superplastic region.\(^3\)\(^1\)\(^\text{11}\)\(^\text{12}\) When threshold stress is taken into consideration, the mechanical properties for superplastic deformation may be described by an equation for the power-law creep of the form\(^12\):

$$\dot{\varepsilon} = \frac{ADGb}{kT} \left( \frac{\sigma - \sigma_{th}}{G} \right)^n \left( \frac{b}{d} \right)^p,$$

where $\sigma_{th}$ is the threshold stress. It has been reported that $n = 2$, $p = 2$, $D = D_2$, in eq. (2) at superplastic deformation in 5083 alloy.\(^3\)\(^1\)\(^\text{13}\)\(^\text{14}\) The relationship between $\dot{\varepsilon}/(D_1)(kT/Gb)$ and $(\sigma - \sigma_{th})/G$ is illustrated in Fig. 2 for the present materials. Table 1 shows the test conditions, the stress exponent, $(\sigma - \sigma_{th})/G$, and the grain size for the materials with the symbols from A to H in Fig. 2. In the superplastic region, threshold stress can be estimated by extrapolation to a zero strain rate of a line, which the data gives as $\sigma$ against $\dot{\varepsilon}^{1/n}$ on a double-linear scale.\(^11\) For example, plots of $\sigma$ against $\dot{\varepsilon}^{1/2}$, $\dot{\varepsilon}^{1/3}$, $\dot{\varepsilon}^{1/5}$ and $\dot{\varepsilon}^{1/8}$ at 803 K for a 5083 alloy with a grain size of 10 $\mu$m are shown in Fig. 3. The linearity of the data in this plot indicates that the presence of a threshold stress is responsible for the change in the strain rate sensitivity exponent, and that the value of $n = 2$ is correct for the stress exponent for superplastic deformation. The same trend was

<table>
<thead>
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<th>Symbol</th>
<th>$T$ (K)</th>
<th>$\dot{\varepsilon}$ (s$^{-1}$)</th>
<th>$(\sigma - \sigma_{th})/G$</th>
<th>$n$</th>
<th>$d$ (µm)</th>
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<tr>
<td>A</td>
<td>803</td>
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<td>$10^{-4}$</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
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<td>$10^{-4}$</td>
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<td>30</td>
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<tr>
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<td>$2.4 \times 10^{-4}$</td>
<td>2</td>
<td>30</td>
</tr>
<tr>
<td>D</td>
<td>803</td>
<td>$10^{-4}$</td>
<td>$3.2 \times 10^{-4}$</td>
<td>2</td>
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<tr>
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<td>$10^{-4}$</td>
<td>$6.8 \times 10^{-4}$</td>
<td>5</td>
<td>30</td>
</tr>
<tr>
<td>F</td>
<td>723</td>
<td>$10^{-3}$</td>
<td>$1.2 \times 10^{-3}$</td>
<td>5</td>
<td>30</td>
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<tr>
<td>G</td>
<td>673</td>
<td>$10^{-4}$</td>
<td>$1.7 \times 10^{-3}$</td>
<td>5</td>
<td>30</td>
</tr>
<tr>
<td>H</td>
<td>673</td>
<td>$10^{-4}$</td>
<td>$1.7 \times 10^{-3}$</td>
<td>5</td>
<td>60</td>
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found in the superplastic region in this investigation. Therefore, all values of threshold stresses in the superplastic region were given by extrapolation to a zero strain rate of a line, which the data gave as \( \sigma \) against \( \dot{\varepsilon}^{1/2} \) on a double linear scale. In the dislocation creep region, the threshold stress was not taken into consideration, and \( \sigma_{th} = 0 \) in Fig. 2. The slope of the line in Fig. 2 decreased as the stress and the strain rate decreased, and the apparent stress exponent, \( n \), changed from 5 to 2. This result indicates that the dislocation-climb is rate-controlling during dislocation creep deformation in the present 5083 alloy. In the superplastic region, the stresses of the alloys differed, depending on the grain size. This result indicates that the transition from dislocation creep to superplasticity occurred as the stress and the strain rate decreased in the present 5083 alloy. The data regarding the present 5083 alloy is summarized together with other reported data\(^{14–17}\). for comparison of superplastic behaviors and for examination of the relationship between \( (\dot{\varepsilon}/D_h)(kT/Gb)(d/b)^2 \) and \( (\sigma - \sigma_{th})/G \), shown in Fig. 4, where \( D_h \) is the lattice diffusion coefficient for pure aluminum.\(^{18}\) It is evident that the relationship between \( (\dot{\varepsilon}/D_h)(kT/Gb)(d/b)^2 \) and \( (\sigma - \sigma_{th})/G \) is represented by a single straight line with a slope of 2 (=n) in the region showing the superplastic flow.

3.2 The relationship between deformation mechanism and GBCD

3.2.1 Stress dependence on GBCD

In order to examine stress dependence on microstructural changes under both superplastic and dislocation creep conditions for 5083 alloy, the changes in GBCD during deformation were investigated. The test conditions can be found in Figs. 2 and 4. The changes in GBCD under superplastic conditions of B, C, and D, and dislocation creep conditions of E, F, and G were investigated. The changes in GBCD in 5083 alloy deformed under superplastic conditions and the dislocation creep conditions are shown in Figs. 5 and 6, respectively. Stress dependence on the GBCD was not observed in the superplastic condition, irrespective of the strain. However, in condition D, although the percentage of random boundaries was unchanged at the initial stage of deformation, the percentage of random boundaries decreased at a strain of 0.6. This finding may be due to grain growth during deformation, which is responsible for the transition of the deformation mechanism from superplastic deformation to slip-creep deformation. Therefore, the GBCD after deformation was almost unchanged, and the percentage of random boundaries was maintained as high as approximately 80% during superplastic deformation.

However, with dislocation creep deformation, the GBCD after deformation changed and the percentage of low-angle boundaries increased with an increase in the strain. Conversely, the percentage of random boundaries decreased with an increase in the strain. The percentage of coincidence boundaries did not change significantly. In addition, stress dependence on the GBCD was observed under dislocation creep conditions. The percentage of increase of low-angle boundaries differed, depending on the amount of stress as well as that of strain. The percentage of increase in low-angle boundaries increased with increases in the flow stress.

3.2.2 Grain-size dependence on GBCD

The grain-size dependence on microstructural change under both superplastic and dislocation creep conditions for 5083 alloy was examined. Under superplastic conditions, changes in the GBCD under the test conditions of A (\( d = 10 \mu m \)) and B (\( d = 30 \mu m \)) in Figs. 2 and 4, respectively, were investigated. The changes in the GBCD under the superplastic conditions are shown in Fig. 7. Grain-size dependence on the GBCD was not observed under the superplastic conditions. The GBCD after deformation was almost unchanged and the percentage of random boundaries reached as high as approximately 80% during superplastic deformation.

On the other hand, in cases involving dislocation creep deformation, stress dependence on the GBCD was observed, such that changes of the GBCD were investigated under conditions of identical stress and identical strain rate. The
changes in the GBCD under the test conditions of G ($d = 30\mu m$) and H ($d = 60\mu m$) were investigated. The fractions of boundaries classified based on coincidence site lattice theory in 5083 alloy deformed under dislocation creep conditions are shown in Fig. 8. The stresses at each test condition were almost identical due to the fact that no grain-size dependence was observed on the flow stress in cases involving dislocation creep deformation, in which grain-size dependence on the GBCD was observed and also in which the degree of increase of low-angle boundaries increased with increases in grain size.

3.3 The relationship between GBS and GBC

The GBC is a very important factor for determining a material’s properties because GBC strongly influences various grain boundary properties. It was found that the microstructure in the present 5083 alloy exhibited various changes under different test conditions or with different grain sizes. The percentage of low-angle boundaries increased with increases in strain during dislocation creep in 5083 alloy. Two possible mechanisms of increasing the percentage of low-angle boundaries with strain can be considered at this point. One mechanism is the absorption of dislocation into random boundaries. The present results suggest that repeated glide-
dislocation pile-up is promoted, as is the absorption of dislocation into random boundaries. Grain boundary dislocations gather glide-dislocations, and then the random boundaries with a misorientation of $\approx 15^\circ$ may change into low-angle boundaries. This mechanism suggests that the development of boundary misorientations enhanced decreasing misorientation between adjacent grains and the percentage of low-angle boundaries increased. However, the percentage of random boundaries with a misorientation of $\approx 15^\circ$ was relatively low using the present materials. Therefore, it is impossible to come to an unambiguous conclusion with this mechanism. Another possible mechanism would be subgrain formation during deformation. McNelley suggested that the development of low-angle boundaries during dislocation creep deformation might reflect subgrain formation. In the present study, the obvious formation of subgrains finer than the initial grains was not observed near the boundaries, as shown by TEM. However, relatively large grains consisting almost of low-angle boundaries were observed by OIM. Figure 9 shows OIM micrographs of the present 5083 alloy deformed to various strains during dislocation creep deformation (test condition F). The measured grain size in the OIM investigation was almost unchanged and was approximately 30 $\mu$m during de-
formation. However, the percentage of low-angle boundaries increased. It is suggested that grain growth occurred and that subboundaries formed in the coarse grains. Therefore, grain growth does not appear to occur, and the relatively large subgrains must have been formed during deformation. Moreover, the percentage of low-angle boundaries increased during deformation. In this manner, these mechanisms, i.e., the absorption of dislocation into random boundaries and subgrain formation, are considered to lead to an increase in low-angle boundaries during dislocation creep deformation. However, additional detailed investigations of this mechanism will still be needed in the future.

It has been reported that GBS was an extremely important mechanism at elevated temperatures and that GBS was influenced by GBC. Kokawa et al. have reported that random boundaries showed a rather larger amount of GBS than did coincidence boundaries in aluminum. It remains possible that GBS influenced microstructural change in the present study. GBS was observed under dislocation creep conditions of 5083 aluminum alloys. The contribution of GBS to total strain at a strain of 0.6 is shown in Fig. 10. These data were obtained from samples designated as E, G, and H in Fig. 2. Under these conditions, the $r_{\text{GBS}}$ in condition E was high, when compared to those in other conditions, as this condition was very close to the superplastic condition. However, the dominant deformation mechanism under condition E was dislocation creep, such that the $r_{\text{GBS}}$ was low, when compared to the reported data of 50–70% under superplastic conditions. A slight decrease in the $r_{\text{GBS}}$ with an increase in flow stress and also with an increase in grain size was observed. This means that the more distant the conditions are from the superplastic condition, the more difficult the GBS. When the results in Figs. 6, 8, and 10 are taken into consideration, it may be concluded that the rate of increase in the percentage of low-angle boundaries was related to the $r_{\text{GBS}}$. The rate of increase of low-angle boundaries increased with decreases in the $r_{\text{GBS}}$ in the dislocation creep regime. Decreasing the $r_{\text{GBS}}$ indicated an increase in the ratio of the contribution of dislocation creep. The movement of glide dislocations was probably promoted by increases in the ratio of the contribution of dislocation creep. The movement of glide dislocations probably promoted GBS; in turn, the occurrence of GBS leads to continuously keeping the high percentage of random boundaries in the superplastic regime. On the other hand, the percentage of random boundaries decreased in the

Fig. 9 OIM micrographs of the present 5083 alloy at strains of (a) $\varepsilon = 0.1$, (b) $\varepsilon = 0.3$ and (c) $\varepsilon = 0.6$. The black line shows boundaries with a misorientation of $\geq 15^\circ$ (random boundaries and coincidence site lattice boundaries) and the gray line shows boundaries with a misorientation of $< 15^\circ$ (low angle boundaries).

Fig. 10 The ratio of contribution of grain boundary sliding at test conditions of E, G and H (in Fig. 2) for 5083 alloy.
dislocation creep regime because GBS rarely occurred. For exhibiting superplasticity, it is necessary to control not only the fine-grained microstructure but also the high percentage of random boundaries during deformation. Therefore, microstructural designs may be necessary to enhance superplastic applications.

4. Summary

(1) There is a close relationship between GBC and the deformation mechanism at elevated temperatures in 5083 alloy. In conditions involving superplastic behavior, random boundaries played an important role during deformation. The percentage of random boundaries was high and did not change during deformation under the superplastic condition. On the other hand, under the slip-creep condition of I/M5083, the percentage of random boundaries decreased and that of low-angle boundaries increased with the progression of deformation. In addition, the degree of increasing low-angle boundaries increased with difference from the superplastic regime.

(2) With superplastic deformation, stress dependence and grain-size dependence on changes in GBC were not observed. However, with creep deformation, these factors were observed. The degree of increase in low-angle boundaries increased with increases in both stress and grain size.

(3) It is possible that GBS influenced microstructural changes during deformation. Random boundaries lead to GBS, and the occurrence of GBS leads to maintenance of a high percentage of random boundaries. In cases involving superplastic deformation, the percentage of random boundaries was high and remained the same during deformation due to continuous GBS. On the other hand, in cases involving slip-creep deformation, the percentage of low-angle boundaries increased with strain, as GBS scarcely occurred. The ratio of increase of low-angle boundaries increased with decreases in \( r_{\text{GBS}} \).

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