Effect of Grain Boundary Characters on Grain Boundary Sliding during Superplastic Deformation

Takumi Haruna†, Toshiya Shibayanagi, Shigenori Hori†‡
and Norio Furushiro

Department of Materials Science and Engineering, Osaka University, 2-1 Yamada-oka,
Suita, Osaka 565, Japan

It is well known that grain boundary sliding (GBS) mainly contributes to superplastic deformation. However, since there are few studies on GBS behavior at each grain boundary during superplastic deformation by using polycrystal material, the contribution of three grain boundary characters to GBS at each grain boundary during superplastic deformation has been investigated. Three characters were adopted as the following: the angle between grain boundary at the specimen surface and tensile direction, the kind of adjacent grains across a grain boundary, and Σ value which was calculated from orientations of adjacent grains across a grain boundary. The examination was carried out by tensile test in SEM with Pb–Sn eutectic alloy at room temperature. The results obtained are summarized as follows:
(1) The superplastic behavior was confirmed to occur when GBS was observed at most of grain boundaries.
(2) It is considered that GBS is easier at a grain boundary both with the higher angle between grain boundary and tensile direction, and with the same kind of adjacent grains across a grain boundary.
(3) So far as Pb–Pb boundary is concerned, GBS was more difficult to occur at grain boundary with rather low Σ value than at random grain boundary. Moreover, for random grain boundary, GBS was easier to occur at a grain boundary with higher angle of grain boundary.

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I. Introduction

It has been shown by numerous studies on superplasticity in fine-grained materials that a double logarithm plot of the flow stress versus the strain rate gives a sigmoidal relationship (19). Superplasticity appears in the intermediate region of the strain rate, so-called "region II", in which the flow stress is most sensitive to the strain rate (30). It has been also clearly shown that a grain boundary sliding (GBS) mainly contributes to the large elongation in the superplastic region (56). Although some mechanisms for the superplastic deformation have been proposed basing on these basic understanding, most of them have little discussion of an elemental process in the microstructural change during the deformation.

Some investigations have been made on GBS since GBS is considered to be an important mechanism in superplastic deformation. In their papers, however, only the kind of the grain boundary was adopted as the grain boundary character (GBC), which is defined by adjacent grains across the grain boundary. At least two other factors should be considered as GBC relating to the GBS;

† Graduate Student, Osaka University. Present address: Department of Materials Science and Processing, 2-1 Yamada-oka, Suita, Osaka 565, Japan.
‡ Present address: Department of Materials Science, Teikyo University, Utsunomiya, Tochigi 321, Japan.
The mean linear intercepts of the grain structure before deformation were 5.0 and 6.4 μm for Sn-rich and Pb-rich phase, respectively.

2. Definition of GBCs and the method of their determination

In this work, as mentioned above, three factors were adopted as the GBCs; the angle between both directions of the tensile axis and of the grain boundary on the surface, which is called hereinafter “the angle of grain boundary”, the kind of adjacent grains across a grain boundary (“the kind of grain boundary”), and “the Σ value” calculated from the orientations of adjacent grains. First two factors were determined by the examination of photographs taken in the SEM before the tensile test. The number of the grain boundaries measured was more than 100 per specimen.

Determination of the orientation for each grain is necessary to calculate the Σ value of each grain boundary. The orientation of such a fine grain of superplastic materials was measured by means of Electron Channeling Pattern (ECP) method. Procedure of the analysis is as follows; area of the self-made ECP stereotriangle coinciding with a ECP photograph of a grain provides the plane orientation and the rotation angle of the grain. A conversion matrix was calculated from both orientations for the adjacent grains. This matrix was compared with the matrix representing the Σ value to obtain a deviation angle of the grain. The Σ value corresponding to the minimum deviation angle by considering the criterion proposed by Brandon as:

\[ \Delta \theta < 15/\sqrt{\Sigma} \]

was adopted as one of GBCs. A grain boundary of the Σ value more than 49 was named as “random” boundary.

In the present study, the ECP analysis was carried out only for the Pb-rich phase due to its crystal structure of FCC.

3. Measurements of distance and direction for GBS

In order to observe the GBS behavior in detail, the specimen surface which was polished and etched was scratched parallel to the tensile direction with a paste including a diamond powder of 1 μm in diameter.

The tensile test in SEM was carried out up to 100% of strain at room temperature. Photographs of the specimen surface were taken at every 10% of strain without stopping the tensile test. Basing on the information from these photographs, both of the distance of GBS and the angle between tensile axis and the direction of GBS were measured for each grain boundary.

III. Experimental Results

1. Deformation characteristics in the ordinary tensile test

Deformation characteristics of the specimen at room temperature were examined by using a tensile testing machine at various strain rates ranging from \(10^{-5}\) to \(10^{-2}\) s\(^{-1}\). In this range of the strain rate, as shown in Fig. 2, the elongation of the specimen increased with decrease in the strain rate, particularly 400% of elongation was obtained at the strain rate of \(1.7 \times 10^{-3}\) s\(^{-1}\). Figure 3 shows the strain rate dependence of both the flow stress at the strain of 10% and the strain rate sensitivity exponent, \(m\) (\(\sigma = K\varepsilon^m\), \(\sigma\): the flow stress, \(\varepsilon\): the strain rate, \(K\): constant). Because of both such low flow stresses and high \(m\) values at slower strain rates, the Sn–Pb alloy used can be realized to exhibit the typical superplasticity at room temperature. According to these results, three typical strain

![Fig. 2 Stress-strain curves of the Sn-Pb eutectic alloy at room temperature and at various initial strain rates.](image)

![Fig. 3 Effect of the initial strain rate on the flow stress at the strain of 10% and the m value.](image)
rates of $8.3 \times 10^{-4}$, $8.3 \times 10^{-5}$ and $4.2 \times 10^{-5}$ s$^{-1}$ were chosen for tensile tests in the SEM.

2. Results of tensile tests in the SEM

1) Distribution of the GBCs

Histograms of the angle and the kind of the grain boundary before deformation are shown in Fig. 4(a) and (b), respectively. It should be noted that the different frequency is found to appear for each GBC. Therefore, the dependence of GBS on GBC has to be discussed under consideration of frequency difference among classes for each character.

As seen in Fig. 4(a), the interval for the angle of the grain boundary was classified by every 10 degree, except for the first interval from 0 to 30 degree, since the number of grain boundaries was much less in this range, so that it was rather difficult to treat statistically these data of this range.

2) Effect of the angle of the grain boundary on GBS

A histogram for the angle of the grain boundary which slides during deformation in the SEM is shown in Fig. 5. The vertical axis in this figure gives the ratio of the number of sliding grain boundaries to total grain boundaries at each class interval of the angle indicated in Fig. 4. The numerals in this figure represent the strain, and the hatched area is the number of the grain boundaries which never slide at all during the deformation up to 100% of strain. As seen in this figure, grain boundaries of higher angles are more facile in sliding than those of lower angles. This behavior is independent of the $m$ value. On the other hand, the hatched area decreased with an increase of $m$ value. In other words, the number of grain boundaries which never slide during the deformation up to 100% decreased under the more superplastic deformation condition. This implies that the uniform sliding of all grain boundaries results in the superplastic deformation.

3) Effect of the kind of grain boundary on GBS

Figure 6 shows a histogram for the kind of sliding grain boundaries. The vertical axis, the numerals, and the hatched area in this figure mean the same as in Fig. 5. This figure gives the fact that GBS occurs more easily at the Pb-Pb and Sn-Sn boundaries than at the Sn-Pb interphase boundary. Moreover, it is recognized that this tendency is independent of the $m$ value, as shown in Fig. 5.

4) Effect of $\Sigma$ value on GBS

A bar chart of $\Sigma$ value with respect to the Pb-Pb boundary at the strain of 10% under the strain rate of $8.3 \times 10^{-5}$ s$^{-1}$ is shown in Fig. 7. The hatched area is the ratio of the number of sliding grain boundaries to the total of grain boundaries. The number of random boundaries is 75% of the total grain boundaries, and about half of these boundaries are found to slide within 10% of strain, while the coincidence boundaries having $\Sigma$ value less than 49 seldom slide in this period. Nevertheless, some of low $\Sigma$ boundaries, for example $\Sigma$ 1 and 3, were found to slide.

Furthermore, the frequency for the angle of the grain boundary is found to make two groups by $\Sigma$ 49, as shown in Fig. 8. Hatched area shows the same meaning as in Fig. 7. In the case of random boundaries shown in Fig. 8(a), the number of the sliding grain boundaries increased with an increase in the angle of grain boundary, while the number of the sliding grain boundaries with less than $\Sigma$ 49 indicated in Fig. 8(b) are independent of the angle of the grain boundary.

IV. Discussion

1. Geometrical relationship between the angle of the grain boundary and GBS

In order to discuss the relation between easiness of
GBS and the angle of the grain boundary, a model is proposed as shown in Fig. 9. The X axis is vertical to the side surface of the specimen, the Y axis parallel to the tensile direction and the Z axis vertical to the top surface of the specimen. It is assumed that a flat grain boundary plane exists in this coordinates as shown in Fig. 9. A vector of vertical to the grain boundary plane is represented as 
\[ [\tan \beta, (\tan \alpha)(\tan \beta), \tan \alpha], \]
where \( \alpha = \) angle OBA and \( \beta = \) angle OBC, while tensile vector is Y direction, i.e., 
\[ [0, 1, 0]. \]
A geometrical sliding direction, BD, on the grain boundary plane is, therefore, defined as 
\[ [(\tan \alpha)(\tan^2 \beta), -((\tan^3 \alpha + \tan^3 \beta), (\tan^2 \alpha)(\tan \beta)), \]
if sliding takes place on the grain boundary plane.

Moreover, a geometrical factor on the grain boundary plane, which is defined as the term 'g', can be determined as "the Schmid factor" from these three directions; the tensile direction, the vertical direction to the grain boundary plane, and the sliding direction on the grain boundary plane. The g factor can be expressed as

$$ g = \sqrt{(x/(1+x))}, \quad x = (1/\tan^2 \alpha) + (1/\tan^2 \beta). $$

(1)

On the other hand, a sliding direction, BE, which is a projection of the sliding direction, BD, on the top of the surface, is as follows:

$$ [(\tan \alpha)(\tan^2 \beta), -(\tan^2 \alpha + \tan^2 \beta), 0]. $$

By introducing the angle between this direction and the tensile direction, defined as $\gamma$, the following equation can be expressed:

$$ [(\tan \alpha)(\tan^2 \beta)]^2 + [(\tan^2 \alpha + \tan^2 \beta)]^2 \cos \gamma = \tan^2 \alpha + \tan^2 \beta. $$

(2)

In practice, the measurable values are angles $\alpha$ and $\gamma$, so that angle $\beta$ can be calculated from eq. (2). Moreover, the g factor of each grain boundary can be estimated from eq. (1).

The relation between the angle $\alpha$ and g factor at each sliding grain boundary can be obtained through the above calculation. In this relation in Fig. 10, the g factor is found to be larger at higher angles. Increasing in g factor means an increase in shear stress on the grain boundary plane. It is suggested, therefore, that the low angle of grain boundaries causes difficulty of GBS, because of the small shear stress on this grain boundary plane.

2. Relationship between GBS and the kind of grain boundary

As mentioned above, most of the papers on the rela-

![Diagram of a sliding grain boundary plane](image-url)
3. Relationship between GBS and $\Sigma$ value

A great deal of studies (12, 13) on the relationship between GBS and the misorientation of adjacent grains have been already carried out mainly for bicrystal specimens. It has been shown in their studies that GBS was rather difficult at low $\Sigma$ grain boundaries while rather easy at random boundaries. Horton and co-workers (14, 16) suggested that a crystal lattice dislocation dissociates into a grain boundary structural dislocation, which move along the grain boundary and also it causes GBS. Kokawa et al. (13) have explained the relationship between GBS and the $\Sigma$ value by observing the grain boundary structural dislocation with TEM. It has been pointed out by them that, at random boundary with low grain boundary energy, GBS occurs easily both because a lattice dislocation dissociates easily into a grain boundary structural dislocation and because the grain boundary structural dislocation is easy to move along the grain boundary. On the other hand, Watanabe (17) has reported the relationship between the intergranular fracture caused by GBS and the $\Sigma$ value for polycrystal specimens. It was found clearly in his work that the intergranular fracture was observed so often at the random boundary, while rarely at the low $\Sigma$ grain boundary. These tendency has been recognized to agree with the present result for the Pb–Pb boundary. Because of the reason previously described, therefore, it has been considered that GBS is difficult to occur at the low $\Sigma$ grain boundary, while easy at the random boundary. However, easiness of GBS has a possibility to be influenced by other GBCs, for example, the angle of grain boundary as well as the $\Sigma$ value, as indicated in Fig. 8.

There is an exception from the above discussion in results of Fig. 7, in which the GBS is shown to occur at very low $\Sigma$ grain boundary, for example, $\Sigma$ 1 and 3. For this point, the more severe analysis for these grain boundaries has been done. The result implies that errors between a deviation angle at them and one calculated from Brandon’s criterion (11) for the $\Sigma$ value are more than 80%. Therefore, since these grain boundaries are found to be rather similar to the random grain boundary, so that this may be caused GBS at the very low $\Sigma$ grain boundary.

It has been suggested that the easiness of GBS is associated with the $\Sigma$ value. However, it is still necessary to obtain the three dimensional information of the grain boundary to understand more fully the GBS behavior during superplastic deformation.

V. Conclusions

The grain boundary sliding behavior during the superplastic deformation of the Pb–Sn eutectic alloy with a fine grain structure has been investigated by in-situ and systematic tensile tests in SEM. Moreover, owing to measurement of the orientation for each grain by means of ECP method, it was possible that each grain boundary was classified according to the $\Sigma$ value. As a result, relationships between GBS and GBCs were understood clear as the following:

1. The superplastic behavior was confirmed to occur when GBS was observed at all of each grain boundary.

2. GBS is considered to take place more easily at grain boundaries both with higher angle to the tensile axis and between the same phases (Sn–Sn, Pb–Pb).

3. It has been clearly shown that the $\Sigma$ value is very possible to influence on GBS; GBS at the Pb–Pb boundary is more difficult at grain boundaries of the low $\Sigma$ value, while, at random grain boundary, it occurs in accordance to the above conclusion of (2).

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