Deformation and Fracture at High Temperatures in an Al–Mg–Mn Alloy Sheet Consisting of Coarse- and Fine-Grained Layers

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High temperature deformation and fracture of the Al–Mg–Mn sheet consisting with the coarse-grained surface and the fine-grained center layers have been investigated. Such a microstructure was produced by the continuous cyclic bending (CCB) and the subsequent annealing. The elongation to failure has a peak value at 713 K at initial strain rates of $5.6 \times 10^{-4} \text{ s}^{-1}$ and $5.6 \times 10^{-3} \text{ s}^{-1}$ in both of as-received sample ($0P$) with fine grains and CCBent and annealed one (20P_A) with the coarse and the fine grains in spite of different microstructures. The $m$ value decreases for 20P_A and increases for 0P with increasing temperature. However, the increase of the $m$ value is not correspondent to the change in the elongation. Deformation mechanism is discussed with activation energy. The SEM micrographs of original surfaces of tensile specimen deformed to failure reveal that at the relatively high temperatures many cracks are formed inside the coarse grains. The features of fractured surface for the 20P_A sample reflect the coarse- and the fine-grained layers faithfully.

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1. Introduction

Fine-grained superplasticity has been vigorously studied in many aluminum alloys more than three decades with incentive to industrial applications. In Class I type alloys such as Al–Mg or Al–Cu base alloys, fairly large elongations have been observed at high temperatures even for coarse-grained samples. It is recently called ‘Class I superplasticity’. This superplasticity has a merit of less cavitation because main deformation mechanism is not grain boundary sliding. On the other hand, Takayama et al. have proposed continuous cyclic bending (abbreviated as ‘CCB’ below) as a strain technique that can produce high strain in the surface layer and low strain in the central part of the metal sheet. These authors reported that the CCB process and subsequent annealing make it possible to produce the gradient microstructure with the coarse-grained surface layer and the fine-grained layer in the middle of the sheet of an Al–4.7 mass%Mg–0.7 mass%Mn alloy (A5083). This cyclic deformation strain technique leads to a definite difference in strain between the surface and central layers. Moreover, the formation of microstructure in the coarse-grained layers was found to be related with sharpening of cube texture.

High temperature deformation of the sheet with the coarse-grained surface and the fine-grained center layers is supposed to show both characteristics of the fine-grained and the Class I superplasticity. Examining the deformation behavior may reveal the difference in mechanism between the two types of superplasticity or contribution of grain boundary sliding. The aim of the present study is to clarify the deformation and fracture at high temperatures in an Al–Mg–Mn alloy sheet consisting of the coarse- and fine-grained layers. Further, deformation mechanism and the effect of temperature on the deformation are discussed.

2. Experimental Procedure

An Al–Mg–Mn alloy sheet of 1.5 mm thick, whose chemical composition is listed in Table 1, was supplied by Sky Aluminum Company, Japan. The as-received sheet was produced through an ordinary industrial process. The sheet had been manufactured by the process of casting, homogenizing at 803 K, hot rolling, cold rolling with a reduction of 75% and a final continuous annealing at 673 K. Workpiece was machined with a 500 mm length, a 20 mm width and a 1.5 mm thickness, and then subjected to CCB using a specially designed device up to the number of passes, $N_{CCB} = 20$. The CCB passes were carried out on both sides of the sheet alternately to keep the symmetry of straining. From the as-received sheet or the CCBent workpiece tensile specimens were machined with a 6 mm length, a 4 mm width and a 1.5 mm thickness of the reduced section, and a 6 mm radius of fillets. A part of these specimens were annealed at 673 K for 3.6 ks in a salt bath. The longitudinal direction of the workpiece, the direction of tension/compression generated on the surface during the CCB and tensile direction of the specimens are parallel to the rolling direction of the as-received sheet.

In the previous study, changes in microstructure during the CCB and annealing were described. Microstructure of the CCBent sample had high-density dislocations in transmission electron micrograph compared with the as-received one. After annealing a coarse-grained structure was observed in the surface layer. The thickness of the coarse-grained layer increased as annealing time or temperature increased. Typical microstructure consisting of the coarse- and the fine-grained layers after CCB and subsequent annealing is shown in Fig. 1. The figure confirms that the microstructure of the

<table>
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<th>Table 1</th>
<th>Chemical composition of alloy used in this study in mass%.</th>
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<td>Si</td>
<td>0.03</td>
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Coarse grained layers

Fine grained layer

500 μm

Fig. 1 Typical microstructure consisting of the coarse- and the fine-grained layers after continuous cyclic bending (CCB) and subsequent annealing. This sample was annealed at 673 K for 120 s after 20-pass CCB.

We used a sample annealed at 673 K for 3.6 ks in a salt bath after 20-pass CCB in the present study. The sample has thickness fraction of 46% for both surface layers with coarse grains, which is called 20P_A below. Grain sizes were measured by the linier intercept method as about 64 μm and about 8 μm for the coarse- and fine-grained layers, respectively in 20P_A. As-received sample, 0P, with a grain size of 7.9 μm was used for comparison to 20P_A. Grain structure of the sample is almost the same as in the fine-grained layer of 20P_A.

Tensile tests were carried out in air in a temperature range of 633–793 K using an Instron type testing machine at an initial strain rate of 5.6 × 10^{-4} – 5.6 × 10^{-3} s^{-1}. The specimens were tested after heating with an infrared furnace at a rate of 0.57 K·s^{-1}. Holding time before tensile testing was 1.8 ks so as to stabilize the test temperature. The elongation to failure was measured from the increase in the gauge length of 5 mm. Original and fractured surfaces of the broken specimens were observed by scanning electron microscopy.

3. Results and Discussion

3.1 Mechanical characterization

Nominal stress vs. strain curves for the as-received (0P) and the CCBent and annealed (20P_A) samples are shown in Figs. 2 and 3, respectively. The curves of 0P show a maximum in stress at an early stage and gradual decrease with strain at each temperature. The maximum stresses for the curves exhibit common temperature dependence of flow stress for high temperature deformation. The maximum elongation to failure of more than 300% is obtained at 713 K in this figure. On the other hand, the curves of 20P_A seem to be similar in shape to those of the 0P at temperatures of 633 K and 673 K, though stress levels are higher by 20%. Different deformation behavior appears at 713 K. The maximum stress is considerably high, which suggests grain coarsening due to holding at a temperature of 713 K being higher than annealing temperature. At and above 753 K, the curves are characterized by a downward convex part after the peak stress at the early stage. The peak stress at 753 K is higher than that at 713 K. The fact also suggests microstructural change in grain size. Elongation to failure shows the maximum value of about 200% at 713 K. This is regarded as the highest temperature at which remarkable microstructural change does not occur.

Figure 4 shows the relationship between the maximum nominal stress and temperature for the two samples. For the 0P sample, the maximum nominal stress decreases with increase in temperature for each strain rate. The relationship between the stress and the temperature can be plotted as a straight line on semilogarithmic scales. The stress lowers to about one tenth at an initial strain rate of 5.6 × 10^{-4} s^{-1} when the temperature is raised from 633 to 793 K, while the stress lowers to about one sixth at 5.6 × 10^{-3} s^{-1}. The stronger dependence of the stress on temperature is found at the lower strain rate. Similar temperature dependence of the stress is observed in
a lower temperature range from 633 to 673 K for the 20P_A sample. However, a slight increase in the stress appears in the range of 713 to 753 K at each strain rate in contrast to 0P. This is attributed to remarkable grain coarsening due to holding at a temperature of 753 K, which is much higher than the annealing temperature of 673 K.

Figure 5 shows elongation to failure as a function of temperature for both samples. The elongation has a peak value at 713 K at both strain rates in both samples. This is almost consistent with the previous result that maximum ductility appeared at 723 K for Al–5 mass%Mg with grain sizes of 40 µm and 90 µm. The difference in elongation between the samples depends on the strain rate. The less difference at the higher strain rate is attributable to the fact that the Class I creep mechanism governing deformation of both samples at the higher strain rate is not strongly affected by grain size. At the lower strain rate ductility of 0P reflects not only the Class I creep but also grain boundary sliding (GBS or fine-grained superplasticity) mechanisms while that of 20P_A results from the former mechanism only.

### 3.2 Strain rate sensitivity

For superplastic materials elongation is often related with strain rate sensitivity \( m \). The maximum stress vs. initial strain rate relationships are displayed at each temperature in Fig. 6. Difference in the stress between 0P and 20P_A is small at relatively low temperatures while the difference is larger at the higher temperatures. There is the larger difference at the lower strain rate clearly. It is confirmed from the stress of the 0P_A sample annealed in the same condition as 20P_A that the annealing has little effect on microstructure in 0P. The results comparatively scattered for the 20P without annealing suggests that stable microstructure was formed in 20P_A by the annealing in a salt bath. The 20P_A sheet consisting
of coarse-grained and fine-grained layers can be supposed as a lamellar composite. The flow stress of coarse-grained layers was calculated from data in 0P and 20P_A using the following equation known as the rule of mixtures for binary composites.9)

\[ \sigma_{20P_A} = \sigma_{CGL} V_{CGL} + \sigma_{FGL} V_{FGL} \]  

where \( \sigma_{20P_A} \), \( \sigma_{CGL} \) and \( \sigma_{FGL} \) are the stresses of 20P_A, coarse- and fine-grained layers, respectively, and \( V_{CGL} \) and \( V_{FGL} \) are the volume fractions of coarse- and fine-grained layers. The stress \( \sigma_{FGL} \) is supposed to equal the stress of 0P, \( \sigma_{0P} \). The fractions \( V_{CGL} \) and \( V_{FGL} \) are replaced by the thickness fractions of 0.46 and 0.54, respectively before the tensile test. Therefore, the fractions \( V_{CGL} \) can be underestimated at and

Fig. 6 Relationship between maximum nominal stress, \( \sigma_{\text{max}} \), and initial strain rate for four types of samples at various temperatures. Solid square means the \( \sigma_{\text{max}} \) of coarse-grained layers estimated according to the rule of mixtures for binary composites under isostress conditions.
above 753 K because of the remarkable grain coarsening as mentioned about Fig. 4. The estimated stress of the coarse-grained layers is also displayed in Fig. 6. The stress of the coarse-grained layers is larger than that of the 0P sample with fine grains. The average ratio $\sigma_{\text{CGL}}/\sigma_{\text{OP}}$ rises from 1.5 at 633 K to 8.3 at 793 K. The estimated stress tends to vary reasonably with temperature although the value at the higher temperatures may include the influence of microstructural change during heating before testing.

Strain rate sensitivity $m$ is plotted in Fig. 7 against temperature for the 0P and the 20P$_A$ samples. The estimated $m$ value for the coarse-grained layers, which was calculated from the slope of each line representing the relation between the estimated stress and the strain rate in Fig. 6, is also shown in the figure. The 0P sample has the $m$ value close to 0.33, indicating deformation by Class I creep, at and below 653 K. Above this temperature the $m$ value increases with temperature up to 0.53 at 793 K in the 0P sample with fine grains. The deformation in this temperature range can be regarded as 'fine grained superplasticity' from the large $m$ value more than 0.37. However, the increase in the $m$ value is not correspondent to the change in the elongation (Fig. 5). On the other hand, the 20P$_A$ sample consisting of the coarse- and the fine-grained layers has a comparatively low sensitivity of strain rate. The $m$ value shows the peak of 0.32 at 653 K, after that it decreases to 0.20, which may be indicative of the rise in the stress due to the microstructural change during heating before testing as described above. The $m$ value estimated for the coarse-grained layer is smaller than that of 20P$_A$. Slight decrease in the $m$ value of the coarse grained layers in the temperature range higher than 700 K can lead to a large drop in the value for the whole sample of 20P$_A$ in spite of the increase in the value for the fine-grained layer.

### 3.3 Temperature dependence

To investigate deformation mechanism for both samples, apparent activation energies $Q_A$ were calculated from relation between strain rate and temperature by setting a constant stress in the stress-temperature plot (Fig. 4). The apparent activation energy of 0P was derived as 112–114 kJ/mol from the relation for a constant stress ranging from 10 to 30 MPa. For 20P$_A$ the activation energy $Q_A$ was 134 kJ/mol for a constant stress of 30 MPa. This value is correspondent to a comparatively low temperature range of 630–690 K, and is fairly close to both activation energies of lattice diffusion of magnesium in aluminum $Q_{\text{Mg}}$ (131 kJ/mol) and lattice self-diffusion in aluminum $Q_{\text{Al}}$ (142 kJ/mol). The Class I creep, which is characterized by stress coefficient $n = 1/m = 3$, is controlled by viscous glide mechanism mainly because solute atoms impede dislocation motion. Therefore if the dominant deformation mechanism is the Class I creep, the relation to $Q_M$ should be discussed. Sherby and Wadsworth found that the activation energy at $n = 2$ was equal to about 135–139 kJ/mol while that at $n = 3$ was equal to about 137–142 kJ/mol in a fine grained Al–5%Mg–1.2%Cr alloy. They concluded that the slip process (dislocation creep) observed in the alloy including second phase particles was identical to that observed in Al–Mg solid solution alloys. For 0P $Q_A$ was much smaller than both the activation energies of lattice diffusion. On the other hand, $Q_A$ for 20P$_A$ was considered to be almost equal to them. Difference between the two samples was attributed to existence of the coarse-grained layers, that is, grain boundary density. Therefore, we understand that the difference was due to degree of contribution of grain boundary diffusion to deformation. It should be noted in the present study that large difference in $Q_A$ appeared between the two samples. The fact can suggest that the surface layers play an important role during deformation, for example on diffusion during deformation.

### 3.4 Scanning electron micrography

Figure 8 shows SEM micrographs of original surfaces of tensile specimen deformed to failure at a strain rate of $5.6 \times 10^{-4} \text{s}^{-1}$ for the 0P sample. Tensile direction is horizontal in the figure. Typical traces of grain boundary sliding are observed at both temperatures. The original surface deformed at 673 K is characterized by granular feature corresponding with a grain size of 7.9 $\mu$m. At the higher temperature of 793 K rows of fibers appear between grains that have original surface before tensile testing. Formation of the rows of fibers was understood as cooperative grain boundary sliding. The difference in the surface feature between at the two temperatures suggests that difference in contribution of grain boundary sliding (GBS) to elongation. The contribution of GBS can not be estimated obviously from these surface features although the GBS is expected to be a dominant mechanism at the higher temperature from the higher m value.

Figure 9 is comparison of the original surfaces at various temperatures and strain rates for 20P$_A$. The surfaces were observed on the coarse grained layers with about a grain size of 64 $\mu$m. Tensile direction is horizontal in the figure also. Openings and steps formed at grain boundaries make it clear that GBS occurred for coarse grains at both strain rates at 673 K (Figs. 9(a) and (d)). Nevertheless the GBS is not main deformation mechanism. In particular, elongated grains at $5.6 \times 10^{-3} \text{s}^{-1}$ (Fig. 9(d)) directly indicate a significant contribution of intragranular slip mechanism. Many cracks are
observed inside the coarse grains at 713 K and $5.6 \times 10^{-4} \text{s}^{-1}$ (Fig. 9(b)), though at this temperature the maximum elongation was obtained for both samples. The cracks seem to intersect trace lines related to rolling. The GBS is supposed to contribute to the elongation at and above 673 K (Fig. 9(a)). Therefore, we can understand that large strain in tensile direction due to the GBS requires a large amount of the intragranular slip as accommodation process, and then such slip leads to cracking. The surface feature at 713 K and $5.6 \times 10^{-3} \text{s}^{-1}$ (Fig. 9(e)) indicates more contribution of the intragranular slips compared with that at $5.6 \times 10^{-4} \text{s}^{-1}$ (Fig. 9(b)). The sufficient contribution results in no cracking inside the coarse grains. On the other hand, few openings at grain boundaries reflect enough contribution of the GBS in contrast to that at 673 K and $5.6 \times 10^{-3} \text{s}^{-1}$ (Fig. 9(d)). When deformed at 793 K, cracking occurs remarkably at $5.6 \times 10^{-4} \text{s}^{-1}$ as shown in Fig. 9(c) because too much GBS takes place to be accompanied with the intragranular slips. An interesting surface is observed at 793 K and $5.6 \times 10^{-3} \text{s}^{-1}$ (Fig. 9(f)). The surface looks to be sheared along the diagonal lines after the elongation due to the intragranular slip mechanism. The shear deformation at the high temperature formed comparatively small grains.

Scanning electron micrographs of fracture surfaces in the 20 A sample are shown in Fig. 10. The sample was tested at the optimum temperature of 713 K. The fracture surface at $5.6 \times 10^{-4} \text{s}^{-1}$ is characterized by the coarsely and the finely ragged features. These features reflect the coarse- and the fine-grained layers faithfully so as to have the same thickness fractions (46% and 54%, respectively as described
Initial strain rate: $5.6 \times 10^{-3} \text{s}^{-1}$, $\varepsilon = 196.6\%$

Initial strain rate: $2.8 \times 10^{-3} \text{s}^{-1}$, $\varepsilon = 183\%$

Initial strain rate: $5.6 \times 10^{-3} \text{s}^{-1}$, $\varepsilon = 211.5\%$

Fig. 10 SEM micrographs representing fractured surfaces characterized by the coarse- and the fine-grained layers in the 20P_A sample. The samples were tested at 713 K.

above). At the higher strain rates fractured surface of the coarse-grained layers necks to the center while fractured surface of the fine-grained layer exhibits similar feature to that at $5.6 \times 10^{-4} \text{s}^{-1}$ except for somewhat narrower thickness. This behavior, necking in the coarse-grained layers, agrees with the results reported in the Class I solid solutions.20

4. Conclusions

High temperature deformation and fracture of the Al–Mg–Mn sheet with the coarse-grained surface and the fine-grained center layers have been investigated. The elongation to failure had the peak value at 713 K in both samples of 0P and 20P_A. The $m$ value decreased with increasing temperature up to 0.20 at 793 K in the 20P_A sample while the $m$ value increased with temperature up to 0.53 in the 0P sample. However, the increase of the $m$ value is not correspondent to the change in the elongation. The SEM micrographs of original surfaces of tensile specimen deformed to failure reveal that at the relatively high temperatures many cracks are formed inside the coarse grains. The features of fractured surface for the 20P_A sample reflected the coarse- and the fine-grained layers faithfully.

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