Superplastic Deformation of AZ61 Magnesium Alloy having Fine Grains

Yu Yoshida1,*1, Keita Araki1,*1, Shota Itoh1,*2, Shigeharu Kamado1 and Yo Kojima2

1Department of Mechanical Engineering, Nagaoka University of Technology, Nagaoka 940-2188, Japan
2President, Nagaoka University of Technology, Nagaoka 940-2188, Japan

Extruded Mg-6 mass%Al-1 mass%Zn (AZ61) alloy was grain-refined utilizing Equal Channel Angular Extrusion (ECAE) processing. Initially, the extruded bar of the alloy was ECAE-processed 2-times at 473 K. Subsequently, it was processed 4-times at 448 K. As a result, the grains are refined to less than 1 μm and a large amount of fine Mg17Al12 compound precipitates. Subsequently, the superplastic properties of the ECAE-processed specimens were investigated. Large fracture elongations of over 300% are obtained at 423 K and 448 K, which is below Tm/2 (Tm: melting point of the alloy), at strain rates above 1 × 10−3 s−1 and 1 × 10−5 s−1, respectively. That is, low temperature superplasticity occurs. Furthermore, at a high strain rate of 1 × 10−3 s−1, superplasticity occurs with the elongation of 242% and 398% at relatively low temperatures of 473 K and 523 K, respectively. In the extraordinarily elongated specimens, significant grain boundary sliding is observed with strain rate sensitivity of 0.3~0.4. The activation energy for superplastic deformation is about 91 kJ/mol, which is close to that for grain boundary diffusion of pure magnesium. It is concluded that the superplastic deformation mechanism of the investigated alloy would be grain boundary sliding accomplished by dislocation slip controlled by grain boundary diffusion.

(Received February 24, 2004; Accepted April 19, 2004)

Keywords: equal channel angular extrusion, AZ61 magnesium alloy, fine grain, superplasticity, grain boundary sliding

1. Introduction

Lightweight magnesium alloys have been attracting attention as environmentally friendly materials due to their desirable properties.1) However, magnesium alloys have some intrinsic problems. Particularly, inferior plastic workability would be a major problem when complex shaped parts are formed at relatively low temperatures. Superplastic forming2–4) is effective for forming complex shaped parts of difficult-to-work materials including magnesium alloys. Superplasticity5–8) generally, occurs at high temperatures or at low strain rates. Therefore, occurrence of low temperature superplasticity at high strain rates is desired for high productivity in case of industrial application. However, there are only a few reports9,10) on the occurrence of low temperature superplasticity at high strain rates in magnesium alloys. It is well known that grain size affects superplastic properties, that is, grain refinement makes it possible for superplasticity to occur easily at low temperatures and at high strain rates.10) In the present study, in order to achieve low temperature superplasticity at high strain rates, extruded bars of Mg-6 mass%Al-1 mass%Zn (AZ61) alloy were subjected to repetitive Equal Channel Angular Extrusion (ECAE) processing11) to refine the grains of the alloy. After that, tensile tests of the ECAE-processed specimens were carried out at the temperature range of 423 K~523 K, which is near Tm (Tm: melting temperature of the AZ61 alloy (893 K12)), under the strain rates of 3 × 10−5~1 × 10−1 s−1. Then the superplastic characteristics and deformation mechanisms were investigated.

2. Experimental Procedure

Extruded bars of Mg-6 mass%Al-1 mass%Zn alloy were machined into cylindrical specimens having a diameter of 15 mm and a height of 80 mm for ECAE processing. The ECAE die used for the present study has two equal channels of 15 mm diameter. The intersecting angle between the two channels is 90° and the angle of the outer arc at the intersection is 60°. Initially, 2-pass ECAE processing was carried out at 473 K. Subsequently, 4-pass ECAE processing was carried out using the same specimen at 448 K to obtain further refinement of the grains. The ECAE specimens were rotated 90° around the longitudinal axis of the specimen after each pass, which is the so-called route Bc, to obtain a homogeneous microstructure. Subsequent annealing was avoided in order to prevent grain growth.

The microstructures of the specimens were observed using optical microscope (OM) and scanning electron microscope (SEM). The etching solution used was 5% picric acid-13% acetic acid-12% distilled water-70% ethanol mixture for OM observation, and 1% nitric acid-20% acetic acid-19% distilled water-60% ethylene glycol mixture for SEM observation. Tensile tests were carried out using as-ECAE-processed specimens. The ECAE processed specimens were machined to JIS H7501 B-type tensile specimen shown in Fig. 1(a). The gauge length of the tensile specimen is 18 mm and the tensile test conditions are shown in Table 1.

Fig. 1 Dimensions of tensile test specimens (a) for investigation of tensile properties and (b) for laser microscopic observation (mm).
The surface roughness of the tensile-tested specimens was measured using laser microscope and compared to that of the tensile specimen before tensile test in order to investigate the contribution of grain boundary sliding to superplasticity. Before tensile test, JIS H7501 S-type plate tensile specimen shown in Fig. 1(b) was highly polished to the average roughness of about 0.02 μm using 0.3 μm and 0.05 μm Al₂O₃ suspension. After that, the specimen was strained, and then the laser microscopic observation was carried out.

3. Results and Discussion

3.1 Microstructures

Figure 2 shows the microstructures of as-received specimen and ECAE-processed specimen. The grains are significantly refined to less than 1 μm and are homogeneously distributed by means of repetitive ECAE processing at 448 K. In addition, the shape of the grains is equixed, indicating that dynamic recrystallization occurs during ECAE processing. SEM photograph of the as-received specimen shows only the existence of some massive Al-Mn compounds. After ECAE processing, numerous fine β phase particles precipitate homogeneously. It is inferred that the fine grains of ECAE specimen are formed by dynamic recrystallization, which is as a result of dislocation pileups that occur in the vicinity of finely precipitated β phase particles. Therefore, AZ61 alloy containing β phase would be more amenable to grain refinement than AZ31 alloy that contains no β phase.13,14)

3.2 Superplastic properties

Figure 3 shows the fracture elongation of ECAE specimens under various test conditions. The elongation increases with decreasing strain rate at each test temperature and increases with increasing temperature at each strain rate, except 3 × 10⁻⁴ s⁻¹. Elongations of more than 300% are achieved at 423 K and 448 K at strain rates slower than 1 × 10⁻⁴ and 1 × 10⁻³ s⁻¹, respectively. Furthermore, the specimen tested at 448 K at 3 × 10⁻⁵ s⁻¹ exhibits extraordinarily large elongation of 1190%. The temperatures of 423 K and 448 K correspond to 0.47Tₘ and 0.50Tₘ, respectively. Thus, in
the investigated alloy, low temperature superplasticity occurs not only at low strain rate range but also at a relatively high strain rate of $1 \times 10^{-3}$ s$^{-1}$ compared to conventional low temperature superplastic materials.$^{15-17}$ In the specimens tested at 473 K and 523 K at the strain rate of $1 \times 10^{-2}$ s$^{-1}$, large elongations of 242% and 398% are respectively obtained. That is, high strain rate superplasticity occurs at such relatively low temperatures. It is noted that the temperatures of 473 K and 523 K at which high strain rate superplasticity occurs correspond to $0.53T_m$ and $0.59T_m$, respectively. They are quite lower temperatures than the superplastic temperatures of conventional high strain rate superplastic magnesium alloys.$^{16,18-20}$ The maximum elongation of the specimen tested at 523 K is smaller than that of the specimens tested at 448 K and 473 K because of grain coarsening. It should also be noted that the tensile specimens used to obtain the large elongations in the present study have a gauge length of 18 mm, which is longer than that of tensile specimens used in previous studies.$^{9,10,15,20}$

Figure 4 shows the outward appearances of specimens after tensile tests. In the specimens tested at lower temperatures and at higher strain rates shown in Fig. 4(b), significant necking occurs, resulting in small elongation. On the other hand, the specimens tested at higher temperatures and at lower strain rates are shown in Figs. 4(c)–(f), uniformly deformed without necking, resulting in large elongations. This is typical superplastic deformation behavior.

Strain rate dependencies of flow stress were investigated in order to evaluate the superplastic deformation mechanism. The results are shown in Fig. 5. The true stress at the true strain $\varepsilon_t$ of 0.2 is used as the value of flow stress $\sigma_f$. The strain rate sensitivity, $m$-value, is calculated according to following equation:

$$m = \frac{\Delta \ln \sigma_f}{\Delta \ln \dot{\varepsilon}}$$

where $\sigma_f$ is the flow stress and $\dot{\varepsilon}$ is the strain rate. As shown in Fig. 5, high $m$-values of 0.3~0.4 are exhibited in the region of superplastic elongation. Generally, the dominant deformation mechanism of fine-grained materials is grain boundary sliding with corresponding $m$-value of about 0.4~0.5.$^{15,16,20,22}$ The $m$-values obtained in the present study are
slightly lower. However, considering the large elongations of the specimens, grain boundary sliding may have contributed to the superplastic deformation.

### 3.3 Superplastic deformation mechanism

#### 3.3.1 Laser microscopic observation

In order to investigate the superplastic deformation mechanism, a tensile specimen was deformed to 100% elongation at 423 K and at a strain rate of $1 \times 10^{-4}$ s$^{-1}$. Before and after the deformation, surface conditions were observed and simultaneously, sectional profiles were measured using laser microscope. Figure 6 shows the laser microscopic image and sectional profile along a line in the image of the specimen (a) before and (b) after deformation. The surface of the specimen before deformation is quite smooth, while the surface of the 100%-deformed specimen has some steps with the height of about 0.2~0.7 µm. As can be seen from Fig. 6(b), the locations of the steps correspond to grain boundaries, indicating the occurrence of grain boundary sliding. Thus, it is confirmed that grain boundary sliding contributes to superplastic deformation in the investigated alloy even at a relatively low temperature of 423 K due to fine grains. However, it can be seen that the grain size in Fig. 6(b) appears to be larger than that of as-ECAE specimen shown in Fig. 2. This suggests that the grains slide aggregately and not individually. Furthermore, the sectional profile is curved in the grain interior, indicating the existence of intragranular deformation. From this observation, it is possible that dislocation slip contributes to deformation as accommodation process.

In Fig. 6(b), it can be seen that a large amount of particles indicated by arrows project out. It is inferred that these particles are $\beta$ (Mg$_{17}$Al$_{12}$) phase. The investigated alloy contains numerous fine $\beta$ phase particles, and it seems that majority of them are not in the grain interiors but on the grain boundaries as can be seen in Fig. 6(b). These facts indicate that the fine $\beta$ phase on the grain boundaries does not obstruct grain boundary sliding. That is, stress concentration near the $\beta$ phase particles by grain boundary sliding would be sufficiently relaxed by diffusion even in the low temperature region below $1/2T_m$.

#### 3.3.2 Estimation of accommodation process

Generally, superplastic deformation is described using constitutive equation for creep as shown below:

$$\dot{\varepsilon} = \frac{AD_0Gb}{kT}(\sigma - \sigma_0)^n(b/\delta)^p \exp\left(-\frac{Q}{RT}\right)$$

where $A$ is a constant, $D_0$ the frequency factor, $G$ the shear modulus, $b$ the Burgers vector, $k$ the Boltzmann constant, $T$ the absolute temperature, $d$ the grain size, $p$ the grain size exponent, $\sigma$ the stress, $\sigma_0$ the threshold stress, $n$ the stress exponent, $Q$ the activation energy and $R$ the gas constant. The activation energy can be evaluated by the following equation when $(\sigma - \sigma_0)/G$ is constant.

$$Q = -\frac{\Delta[\ln(\dot{\varepsilon}/G)(d/b)^p]}{\Delta[1/(RT)]}$$

The threshold stress $\sigma_0$ was evaluated by double linear plot of $\sigma$ against $\dot{\varepsilon}^{1/n}$. The best linear fit is obtained at $n = 2$ as shown in Fig. 7. Then the $\sigma_0$, indicated in Fig. 7, was determined from $\sigma$ value at zero strain rate. The grain size

Fig. 6 Laser microscopic images (top) and sectional profile (bottom) of undeformed specimen (left) and specimen deformed 100% at 423 K under a strain rate of $1 \times 10^{-4}$ s$^{-1}$. Gray scale in the laser microscopic image indicates height of the observed plane.

Fig. 7 Double linear plots of $\sigma$ against $\dot{\varepsilon}^{1/n}$ ($n = 2$).
Grain refinement of extruded AZ61 magnesium alloy was carried out utilizing repetitive ECAE processing. Superplastic properties of the obtained specimens and their deformation mechanism were investigated. As a result of combined 2-pass and 4-pass ECAE processing at 473 K and 448 K, respectively, a large amount of β phase precipitates and simultaneously, the grains are significantly refined to less than 1 μm. During tensile tests of the ECAE-processed specimens, low temperature superplasticity occurs with elongations of over 300% at a relatively high strain rate of $1 \times 10^{-3} \text{s}^{-1}$. Furthermore, high strain rate superplasticity occurs at 473 K and 523 K, which are sufficiently lower temperatures than the superplastic temperatures of conventional high strain rate superplastic materials. The specimens in which superplasticity occurs exhibit strain rate sensitivity of 0.3–0.4, suggesting that grain boundary sliding is the dominant deformation mechanism. In fact, the activity of grain boundary sliding is confirmed even at a relatively low temperature of 423 K. The activation energy for superplastic deformation is about 91 kJ/mol, which is close to that of grain boundary diffusion of pure magnesium. Therefore, slip deformation controlled by grain boundary diffusion contributes to deformation as an accommodation process.

Acknowledgements

The present study is supported by COE Program for the 21st Century “Creation of Hybridized Materials with Superfunctions and Formation of International Research & Education Center” from Nagaoka University of Technology.

REFERENCES