Low Temperature Superplasticity of ECAE Processed Mg–10%Li–1%Zn Alloy

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In order to achieve low temperature superplasticity at relatively high strain rate in magnesium alloys, Mg–10 mass%Li–1 mass%Zn (LZ101) $\alpha + \beta$ two phase alloy was subjected to ECAE (Equal Channel Angular Extrusion) processing and tensile tests of the obtained specimens of the alloy were carried out to investigate the superplastic properties. In a specimen of the LZ101 alloy, which has improved microstructure through repetitive ECAE processing at 323 K, superplasticity occurs at 423 K, which is below $T_m/2$ (T_m : melting point of the alloy), under a relatively high strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ with fracture elongation of 391%. Such a specimen after tensile test contains fine grains due to dynamic recrystallization and the precipitation of β phase along the grain boundaries and at triple points in recrystallized α phase. This microstructural change enhances grain boundary sliding, resulting in the occurrence of low temperature superplasticity at relatively high strain rate.

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1. Introduction

Recently, housings of electronic appliances, such as notebook computers and mobile phones, are being made lighter and more compact. Therefore, materials for such applications require not only high strength but also high ability to dissipate heat together with superior damping capacity, good electromagnetic shielding characteristics and good recyclability. Magnesium alloys have been attracting attention as the suitable materials that could satisfy the above requirements. However, inferior plastic workability at room temperature remains the major problem of these alloys. The housings of electronic appliances have many complex shaped parts such as boss, rib and hinge, therefore, superplastic forming is important for their production process in order to obtain near-net shaped products using magnesium alloys. Unfortunately, superplasticity usually occurs at high temperatures or under low strain rates. However, the occurrence of low temperature and high strain rate superplasticity is preferred due to cost and productivity considerations. Previous works^{1–10)} have indicated that superplasticity occurs in magnesium alloys at low temperatures or under high strain rate by grain refinement, however, there are only few reports^{11,12)} on the occurrence of low temperature superplasticity at high strain rates.

It is well known that addition of lithium to magnesium tremendously improves plastic formability of magnesium alloys at room temperature because apart from magnesium solid solution (α phase), lithium solid solution (β phase) that has bcc crystal structure also crystallizes if 5.7 mass% or more of lithium is added.^{13–15)} In a previous study,¹⁶⁾ it was found that low temperature superplasticity occurs at 373 K under a relatively low strain rate of about 10^{-4} s^{-1} in ECAE (Equal Channel Angular Extrusion)^{17,18)} processed Mg–Li–Zn $\alpha + \beta$ two phase alloys which have very fine grains. This temperature is among the lowest temperatures in the previous results on superplasticity of Mg–Li–Zn $\alpha + \beta$ two phase alloys. However, the strain rate remains low. In the present study, in order to achieve low temperature su-

perplasticity at higher strain rates, cast specimens of Mg–10.2 mass%Li–0.8 mass%Zn (LZ101) alloy were subjected to repetitive ECAE processing to improve their microstructure. Furthermore, the obtained specimens were subjected to tensile tests at the temperature range of $0.43\text{--}0.51T_m$ (T_m : melting temperature of the Mg–10 mass%Li alloy (863 K¹⁹⁾)) under the strain rates of $3 \times 10^{-5}\text{--}1 \times 10^{-2} \text{ s}^{-1}$. The relationship between tensile test conditions and superplastic characteristics was also investigated.

2. Experimental Procedure

Melting of the LZ101 alloy used in this study was conducted in vacuum high frequency induction furnace in argon atmosphere without using any flux. Then cylindrical specimens having a diameter of 15 mm and a height of 80 mm for ECAE processing were machined from the obtained ingot. First, in order to achieve grain refinement and spheroidization of α phase, the specimens were subjected to 2-pass ECAE processing at 323 K with rotating angle of 180° around the longitudinal axis of the specimen and subsequently annealed isochronally at 623 K for 1 hour. α phase is expected to recrystallize during annealing at that temperature. Furthermore, this specimen was subjected to 4-pass ECAE process at 323 K with rotating angle of 180° , 90° and 180° for each pass to introduce a large amount of strain in the specimen. This 6-pass ECAE-processed specimen was not annealed because it has been established that dynamic recrystallization occurs in the investigated alloy during tensile test¹⁶⁾ and results in superplasticity.

The microstructures of the obtained specimens were observed on planes parallel to the extrusion direction using optical microscope. The specimens were etched using 10% HNO_3 solution in ethanol. Tensile tests were carried out using 6-pass as-ECAE-processed specimens that were extracted such that the tensile direction was parallel to the extrusion direction. Figure 1 shows the dimensions of tensile test specimen, while the tensile test conditions are shown in Table 1. After tensile test, the specimens were polished and etched us-

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Fig. 1 Dimensions of tensile test specimen.

Table 1 Tensile test conditions.

Specimen	6-pass as-ECAE			
Initial strain rate, $\dot{\epsilon}/\text{s}^{-1}$	3×10^{-5}	1×10^{-4}	1×10^{-3}	1×10^{-2}
	1×10^{-5}	1×10^{-4}	1×10^{-3}	1×10^{-2}
Test temperature, T/K	373	398	423	448

*The test was carried out only at 373 K.

ing 5% picric acid-13% acetic acid-12% distilled water-70% ethanol mixture. Then the microstructures near the fractured surfaces and the uniformly elongated region of the fractured specimens were observed using high resolution digital microscope in order to investigate the dominant deformation mechanism. Furthermore, the microstructures of the uniformly elongated region of the fractured specimens were also observed using scanning electron microscope (SEM) under acceleration voltage of 15 kV.

3. Results

3.1 Microstructures

Figure 2 shows the microstructures of as-cast, 2-pass ECAE, and 6-pass ECAE-processed specimens. The microstructure of the specimen annealed at 623 K for 1 h after 2-pass ECAE processing is also shown. Needlelike α phase is observed in the as-cast specimen. In the 2-pass ECAE-processed specimen, the α phase appears to be elongated to a specific direction, and the β phase exhibits a deformed structure as a result of severe working during ECAE processing at 323 K. The α phase undergoes both refining and spheroidaizing, and the grains of the β phase are coarsened by subsequent annealing at 623 K for 1 h as shown in (c). After the annealed specimen was subjected to further 4-pass ECAE processing, both α and β phases show a deformed structure, indicating that a large amount of strain has been introduced in the specimen as shown in (d). Some fine and equiaxed grains are also observed in the β phase as shown in (e), which is a higher magnification of (d). It might be said that these fine and equiaxed grains are dynamically recrystallized during ECAE processing at 323 K, because it has been suggested that the recrystallization temperature can be reduced to 323 K in highly strained 4-pass ECAE-processed Mg-10%Li-1%Zn alloy.²⁰⁾

3.2 Tensile properties

Figure 3 shows the true stress-true strain curves of the 6-pass ECAE-processed specimens tested at 373–448 K under an initial strain rate of 3×10^{-5} – $1 \times 10^{-2} \text{ s}^{-1}$. The tensile properties exhibit significant strain rate dependencies at all test temperatures and the flow stresses increase with increasing strain rate and decreasing test temperature. Furthermore,

Fig. 2 Microstructures of (a) as-cast, (b) 2-pass ECAE-processed, (c) subsequent annealing at 623 K for 1 h and (d and e) 6-pass ECAE-processed specimens.

Fig. 3 True stress-strain curves of 6-pass ECAE-processed specimens.

the flow stresses decrease with increasing strain during tensile test. The decrease in flow stress becomes gradual and significant strain hardening occurs with increasing test temperature and as the strain rate decreases. The specimens experienced large prestrain before tensile test, and therefore, the specimens tested under a high strain rate of $1 \times 10^{-2} \text{ s}^{-1}$ at relatively low temperatures of 373 and 398 K do not exhibit strain hardening, and their flow stresses decrease suddenly with necking after maximum tensile strength. On the other hand, strain hardening occurs easily under a low strain rate of $1 \times 10^{-4} \text{ s}^{-1}$ at 423 and 448 K because the prestrain is easily

released at the beginning of tensile test. In addition, specimens tested at low strain rate conditions indicate a steady state flow region. This suggests that the progress of necking is extremely gradual in those specimens. It is thought that dynamic recovery or recrystallization occurs and deformation progresses with balancing of work hardening in the steady state flow region. These stress-strain curves are typical for superplastic deformation,^{2,6)} and the specimens exhibit large fracture elongation.

3.3 Strain rate dependencies of tensile properties

Figure 4 shows strain rate dependencies of tensile properties of the investigated alloy. The true stress corresponding to true strain of 0.2 was used as the flow stress. The strain rate sensitivity, m -value, was evaluated from double logarithm plotting of the relationship between strain rate and flow stress in order to investigate the occurrence of superplasticity, and the results are also shown in Fig. 4. High fracture elongation of over 200% are observed with high m -values of 0.3–0.5 at all temperatures in the strain rate range up to $1 \times 10^{-3} \text{ s}^{-1}$. Thus, superplasticity occurs even at low temperatures below $0.5T_m$ in 6-pass ECAE-processed LZ101 alloy.

In the specimen tested at 373 K, the elongation reaches 421% at an initial strain rate of $3 \times 10^{-5} \text{ s}^{-1}$, and the m -value is 0.5, which is the highest value among all test conditions. Both the m -value and the elongation decrease with increasing strain rate. Furthermore, a peak of elongation does not appear under the investigated strain rate range. Therefore, it is expected that higher elongation can be obtained if tensile test is carried out under slower strain rates than $3 \times 10^{-5} \text{ s}^{-1}$.

In the specimen tested at 398 K, although maximum elon-

gation is smaller than the specimen tested at 373 K, it shows an elongation of 313% under a relatively high strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. It appears that a peak of elongation exists between the strain rates of 1×10^{-4} and $1 \times 10^{-3} \text{ s}^{-1}$ since the value of elongation is same at both strain rates.

In the specimen tested at 423 K, the elongation obtained under the strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ increases further than that of the specimen tested at 398 K and the value is 391%. The specimen also shows a high m -value of 0.4. This temperature corresponds to $0.49T_m$, and the strain rate is higher than that for conventional low temperature superplasticity.^{2–7)} This implies that low temperature superplasticity occurs at 423 K at a relatively high strain rate in the investigated alloy. Furthermore, an elongation of 206% is obtained even at a high strain rate of $1 \times 10^{-2} \text{ s}^{-1}$. However, elongation decrease at all strain rates in the specimen tested at 448 K compared with the specimen tested at 423 K.

4. Discussion

Microstructures of the tensile tested specimens were observed in order to reveal the deformation mechanisms during tensile test. Figure 5 shows microstructures near the fractured surfaces (A) and those of the uniformly elongated region (B) of the specimens after tensile test. Similarly the microstructure at position (B) was observed before tensile test and it is also shown in the figure. The microstructure of the specimens of (c) and (d) tested under $1 \times 10^{-2} \text{ s}^{-1}$ at 373 K and 423 K respectively indicate that local deformation at the region near the fracture surface contributes to fracture elongation of these specimens, and this agrees with the stress-strain curves shown in Fig. 3. On the other hand, in the specimens of (a) and (b) that exhibit extraordinary elongation of 421 and 391%, respectively, α phase particles are elongated to the tensile direction in the overall gage section. Despite this huge deformation, grains of the superplastically deformed specimens are equiaxed for both α and β phases. These specimens also show high m -values of about 0.4 or 0.5 respectively. From these results, it is considered that superplastic deformation of 6-pass ECAE-processed specimens of LZ101 alloy that experiences a large amount of prestrain before tensile test result from grain boundary sliding that is enhanced by fine grains dynamically recrystallized during tensile test. Furthermore, some voids are observed at $\alpha - \beta$ boundaries. This indicates that the fracture of these specimens occurs by growth and interlinkage of the voids at $\alpha - \beta$ boundaries.

It is likely that the deformation is more difficult to occur in α phase than in β phase due to the hcp crystal structure of the former. Therefore, it is suggested that α phase may be an obstacle to obtain large superplastic elongation in high β -containing $\alpha + \beta$ two phase alloys. In the superplastic deformation of an $\alpha + \beta$ two phase alloy, it is important that α phase also deform largely with flow of the β phase because voids are often generated at $\alpha - \beta$ boundaries. The very fine grains observed in the investigated alloy would be responsible for enhanced grain boundary sliding in α phase. Figure 6 shows SEM images of the uniformly elongated regions (B) of specimens that are shown in Fig. 5. In the specimens that exhibit extraordinary elongation of 421% and 391%, (a) and (b), respectively, some black particles are observed in the gray α

Fig. 4 Strain rate dependencies of (a) flow stress under true strain of 0.2 and (b) fracture elongation. Strain rate sensitivity exponent, m -values, are also indicated in (a).

Fig. 5 Microstructures near the fractured surfaces (A) and uniformly elongated region (B) of specimens after tensile test. (a) 373 K, $3 \times 10^{-5} \text{ s}^{-1}$ ($e_f = 421\%$), (b) 423 K, $1 \times 10^{-3} \text{ s}^{-1}$ ($e_f = 391\%$), (c) 423 K, $1 \times 10^{-2} \text{ s}^{-1}$ ($e_f = 206\%$), (d) 373 K, $1 \times 10^{-2} \text{ s}^{-1}$ ($e_f = 101\%$).

phase. According to the phase diagram of Mg–Li binary alloy,¹⁹⁾ it is considered that these particles are β phase that precipitated during tensile test from super-saturated α phase generated by intermediate annealing at 623 K. These β phase particles are also observed in Figs. 5(a) and (b) as indicated by arrows, and it appears they precipitate along grain boundaries and at triple points in the α phase. On the other hand, in the specimens tested under $1 \times 10^{-2} \text{ s}^{-1}$, the precipitation of the β phase is not observed as shown in Figs. 6(c) and (d). Furthermore, precipitation of the α phase in the β phase is observed in all specimens as shown in Fig. 5. The volume fraction of the β phase in the specimens that exhibits extraordinary elongation is relatively higher than in the specimens that show low elongation. It has been reported²¹⁾ that super-plastic elongation increases with increase in volume fraction of β phase in $\alpha + \beta$ phase titanium alloys. Therefore, it is considered that the precipitated β phases contribute to super-

Fig. 6 SEM images of specimens after tensile test. (a) 373 K, $3 \times 10^{-5} \text{ s}^{-1}$ ($e_f = 421\%$), (b) 423 K, $1 \times 10^{-3} \text{ s}^{-1}$ ($e_f = 391\%$), (c) 423 K, $1 \times 10^{-2} \text{ s}^{-1}$ ($e_f = 206\%$), (d) 373 K, $1 \times 10^{-2} \text{ s}^{-1}$ ($e_f = 101\%$).

Fig. 7 Schematic illustration of mechanism for superplastic deformation.

plastic deformation.

From the above considerations, the mechanism for super-plastic deformation in the investigated alloy can be illustrated as shown in Fig. 7. The grains in both α and β phases are recrystallized at the first stage of tensile deformation due to high strain that accumulated during ECAE processing, and the fine recrystallized grains increases the grain boundary area, thereby enhancing grain boundary sliding. As observed earlier, in the α phase, the β phase particles that have high deformability precipitate along the grain boundaries and at triple points, which serve as favorable nucleation sites for the recrystallized grains. Therefore, it might be expected that the β phase particles precipitated in α phase would contribute to relax the stress concentration by enhancing grain boundary sliding in addition to accommodation process in α phase. Under the presence of very fine β phase particles on grain boundaries of α phase, large elongation can be obtained in the investigated alloy even at low temperatures below $0.5T_m$ at relatively high strain rates. Under the high strain rate of $1 \times 10^{-2} \text{ s}^{-1}$, recrystallization is difficult and the β phase particles do not precipitate in the α phase probably due to short test time as shown in Figs. 6(c) and (d). This makes accommodation process more difficult, and as a result, fracture elongation is expected to be small.

Furthermore, in the specimens that exhibit extraordinary

elongation of 421% and 391%, the α phase breaks up as shown in Fig. 5. It is inferred that this results from grain boundary sliding and the precipitation of β phase in the α phase according to the mechanism illustrated in Fig. 7.

5. Conclusions

In this study, ECAE process was applied to $\alpha + \beta$ two phase LZ101 alloy in order to achieve low temperature superplasticity at relatively high strain rates by improvement of its microstructure. Furthermore, strain rate dependencies of the tensile properties and the mechanism of superplasticity were investigated. A summary of the results is described as follows:

(1) Refining and spheroidizing of α phase is achieved in LZ101 alloy by applying repetitive ECAE processing at 323 K and subsequent isochronal annealing at 623 K for 1 h. Furthermore, a large amount of strain is accumulated in the β phase by applying further 4-pass ECAE processing at 323 K.

(2) The tensile properties of 6-pass ECAE-processed LZ101 alloy show strain rate dependencies at all test temperatures of 373–448 K and the strain rate sensitivity, m , of 0.3–0.5 is obtained under the strain rate range up to $1 \times 10^{-3} \text{ s}^{-1}$. Superplasticity occurs in these specimens, especially, at 423 K that correspond to $0.49T_m$ of the alloy. An extraordinary elongation of 391% is obtained with m -value of 0.4 at a relatively high strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ with gradual necking. Furthermore, large elongation of 206% is obtained even at a high strain rate of $1 \times 10^{-2} \text{ s}^{-1}$.

(3) In the specimens where superplasticity occurs, fine grains that are generated by dynamic recrystallization during tensile test enhance grain boundary sliding in both α and β phases. Furthermore, in the α phase, β phase particles precipitate along the grain boundaries and at triple points, thereby contributing to accommodation process. Therefore, low temperature superplasticity occurs at relatively high strain rate in the investigated alloy.

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