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Development of High Strength and Toughness Magnesium Alloy by Grain Boundary Control

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Abstract

The mechanical properties, such as strength and fracture toughness, were investigated using caliber rolled Mg-6wt.%Al-1wt.%Zn (AZ61) alloy, which is the material consists of a constrained plane other than the rolling direction plane and is compressed non-simultaneously from two directions. The initial microstructural observations showed that the caliber rolled AZ61 alloy had a high fraction of low-angle grain boundaries and an average grain size of 2.2 μm. In addition, particles with an average size of 100 nm existed in the matrix. This alloy showed a yield strength of 423 MPa and a fracture toughness of 34.1 $MPam^{1/2}$. A combination of grain refinement, formation of low-angle grain boundaries and dispersion of fine particles is one of the effective microstructural controls to produce the magnesium alloys with fracture toughness similar to the conventional high strength aluminum alloys.

Introduction

Magnesium alloys are the lightest among all the structural alloys in use, but their fracture toughness, which is one of the mechanical properties for the judgment of reliability and safety requirement, has generally been reported to be lower than that in aluminum alloys [1,2]. The lower fracture toughness in magnesium results from the deformation twins, which form at the very beginning of plastic deformation [3]. Since the accumulated plastic strain by the twins is smaller than that of the grain boundary migration by the dislocation slip, the boundaries around twins define the crack propagation route [4]. On the contrary, the deformation twins are reduced or prevented by refining the structure [5], and thus, the fracture toughness tends to increase with grain refinement [6].

Some reports recently pointed out that the formation of a low-angle grain (or sub-grain) boundary structure is effective for the enhancement of ductility in metallic materials [7-9]. Although the grain boundary generally prevents the dislocation movement, it provides the nucleation site for the micro-void and/or -crack formation in the plastic deformation. On the other hand, the amount of dislocation pile-up at the low-angle grain boundaries is lower than that at the high-angle grain boundaries, leading to an advantage towards ductility. Our earlier study has reported the effect of low-angle grain boundaries on the mechanical properties and fracture in magnesium alloy [10]. The previous results showed that the fine-grained Mg-3wt.%Al-1wt.%Zn (AZ31) alloy with a high fraction of low-angle grain boundaries enhanced the fracture toughness, and these boundaries did not become the origin of the micro-void formation.

Particle dispersion is also recognized as an influential microstructural factor because of being the void nucleation site [11,12]. Since finer particles are more difficult to become the void nucleation site compared to coarser particles, control of the particle size, i.e., refinement of particle size, is one of the useful methods for the improvement of fracture toughness. Thus, the combination of grain refinement, high fraction of low-angle grain boundaries and a dispersion of particles has the potential to produce a magnesium alloy with high strength and fracture toughness; however, there are no reports. Therefore, the initial microstructures and mechanical properties were investigated by using a Mg-6wt.%Al-1wt.%Zn alloy, which was produced by caliber rolling, in this study.

Experimental Procedure

A commercially extruded rod of Mg-6wt.%Al-1wt.%Zn (AZ61) alloy with a diameter of 42 mm was used in the present study. The billet was kept at a temperature of 473 K for 30 minutes in a furnace, and then subjected to plastic working by the caliber roll at room temperature. The billet was passed through 14 times and twice for the final groove to control the cross-sectional shape of the bar, and then was quenched in water. After rotating the rolled billet 90° clockwise around the rolling axis, the billet is subjected to rolling in the same way as the former pass. The detailed experimental procedure is reported elsewhere [13]. The caliber rolled AZ31 alloy was also prepared by the same procedures as AZ61 alloy [10].

The initial microstructure of the rolled alloy was observed by the electron back-scattered diffraction (EBSD) and transmission electron microscopy (TEM). The observed area in the initial sample was the RD-TD plane. The tensile test was performed at an initial strain rate of 1×10^{-3} s^{-1} at room temperature. Tensile specimen with a gauge length of 15 mm and a gauge diameter of 3 mm was machined from the rolled bar to make the tensile axis parallel to the rolling direction.

Fracture toughness was measured using the *J*-integral method according to the ASTM E1890 [14]. The specimen that was used for investigating the fracture toughness consisted of a three point bending sample with a width of 10 mm, a thickness of 5 mm and a length of 44 mm. The specimen was machined directly from the rolled bar, and a V-notch was made normal to the rolling direction. The fracture surface was also observed by scanning electron microscopy (SEM) after the fracture toughness test in the ASTM E399 [15].

Results and Discussion

Typical initial microstructure by EBSD observation in caliber rolled alloys is shown in Fig. 1: (a) inverse pole figure image and (b) distribution of misorientation angle. The average grain size, which consisted of the high-angle grain boundary ($\theta \geq 15°$), was obtained to be 2.2 ± 0.5 μm by EBSD measurement. Figure 1(b) shows that the fraction of grain structures with the misorientation angles of $5° \leq \theta < 15°$, i.e., low-angle grain boundary, was obtained to be 0.25 in the present alloy. This figure also includes the result of the AZ31 alloy, which was produced by the same process [10]. Both the alloys show a similar distribution of misorientation angle, and thus, they are found to have a similar fraction of low-angle grain boundaries. The bright field image of the AZ61 alloy is shown in Fig. 2. The grain size was ~ 3 μm, which is the same as the EBSD observation. The existence in the spherical shaped particles with an average size of 100 nm is observed in the matrix. The particle was identified as the β-phase ($Mg_{17}Al_{12}$) by EDS analysis.

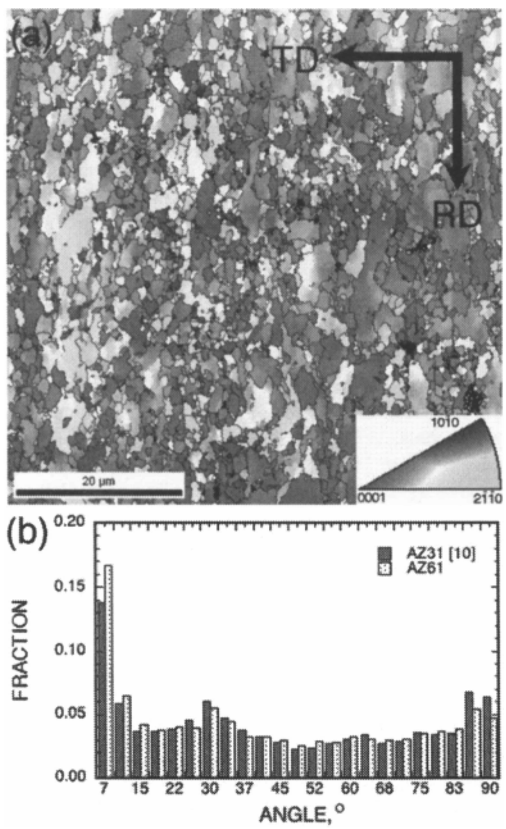

Fig. 1: Typical initial microstructures by EBSD observation in the caliber rolled alloys: (a) inverse pole figure and (b) distribution of misorientation angle. The black lines in Fig. (a) are the high-angle grain boundary ($\theta \geq 15°$), and Fig. (b) includes the previously reported data of the caliber rolled AZ31 alloy [10].

The mechanical properties of the AZ61 alloy are summarized in Table 1. Since the fracture toughness in the as-received commercially AZ61 alloy is around 23 $MPam^{1/2}$, the fracture toughness is improved by the caliber rolling. This table includes the data for the caliber rolled AZ31 alloy [10]. The present caliber rolled AZ61 alloy also shows higher strength and fracture toughness balance compared to those in the AZ31 alloy. In addition, the fracture toughness of the conventional aluminum alloys, i.e., 7xxxAl alloys, are reported to be ~ 35 $MPam^{1/2}$ [16], which is found to be similar to the present AZ61 alloy.

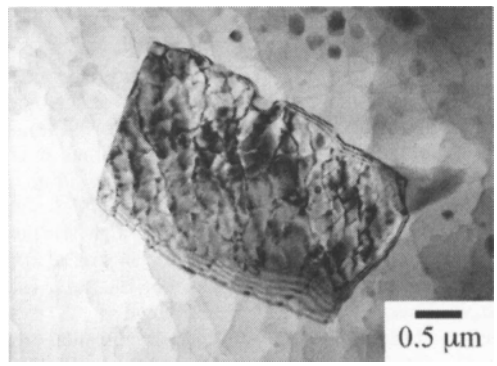

Fig. 2: Bright field image of the caliber rolled AZ61 alloy.

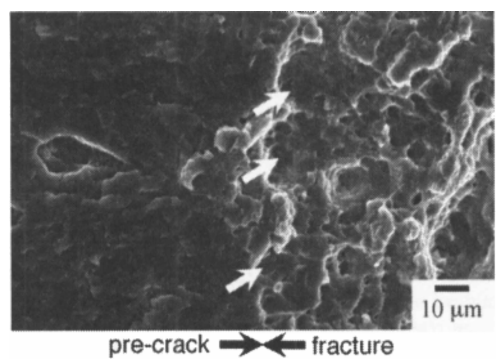

Fig. 3: Typical fracture surface by SEM observation. White arrows mark the dimples.

Hereafter, the reason for the higher fracture toughness in the present alloy is discussed by a comparison between the caliber rolled AZ31 and AZ61 alloys, which have high fraction of low-angle grain boundaries. The grain size is one of the most influential microstructural factors for the fracture toughness; however, the AZ31 and AZ61 alloys have similar sizes of 2 ~ 3 μm. The fracture toughness is reported to be proportional to one-third power of the concentration of solid solute atoms: $\Delta K_{IC} \propto C^{1/3}$ [17]. When the additional concentration of solid solute atom, i.e., aluminum, is 3 wt.%, the improvement for fracture toughness, ΔK_{IC}, is estimated to be ~ 2 $MPam^{1/2}$. However, this estimated value of ΔK_{IC} is smaller than that in the increment of 5 $MPam^{1/2}$ in the experimental result. Thus, the increment in the fracture toughness in the AZ61 alloy is due to not only the solid solution strengthening but also the other microstructural factors.

Typical fracture surface by SEM observation is shown in Fig. 3. This figure shows that there are many dimples, which are typical of a ductile fracture pattern, marked by white arrows.

The dimple nucleation originates from the coarse particle, and the fracture occurs at the interface between the matrix and the particle [18] or in the particle-itself [19]. A small amount of the dimple nucleation is observed in this figure due to no existence of coarse particles. Thus, the dimple nucleation site is assumed to the presence of particle at the grain boundaries. On the contrary, the fracture toughness increases with the dispersion of fine particle, since they act as obstacles for the dislocation movements around them. Our previous report shows creation of a dislocation net-work around fine particles by the deformed microstructural observations, and predicts the increment ratio of fracture toughness based on Orowan strengthening mechanism [20]. The increment ratio for fracture toughness is estimated to be ~ 10 %, when spherical shaped particles with an average size of 100 nm are dispersed into the matrix. The AZ61 alloy contains particles, as shown in Fig.2, but there are no particles in the caliber rolled AZ31 alloy [10]. Thus, the AZ61 alloy is estimated to show 10 % higher fracture toughness, which corresponds to ~ 3 MPam$^{1/2}$, than that in the AZ31 alloy; the sum of the slid solution strengthening and the dispersion of spherical shaped fine particle is in agreement with the experimental result of 5 MPam$^{1/2}$. Therefore, the superior mechanical properties in the present alloy are related to not only the high fraction of low-angle grain boundaries but also the dispersion of the fine precipitate particles into the fine grains.

Summary

1) The initial microstrucutal observations showed that the caliber rolled AZ61 alloy had an average grain size of 2.2 μm and high fraction of low-angle grain boundaries. The β-phase particles with an average size of 100 nm were dispersed into the matrix.
2) The caliber rolled AZ61 alloy showed a balance of high strength and fracture toughness, which was the similar to the conventional high strength aluminum alloys.

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Table 1: List of the mechanical properties in the caliber rolled AZ31 [10] and AZ61 alloys.

material	d, μm	σ_{ys}, MPa	σ_{UTS}, MPa	δ, %	K_{IC}, MPam$^{1/2}$	
AZ61	2.2	423	438	9.4	34.1	this study
AZ31	2.5	369	385	10.9	29.4	[10]

where d is the grain size, σ_{ys} is the yield strength, σ_{UTS} is the ultimate tensile strength, δ is the elongation-to-failure and K_{IC} is the plane-strain fracture toughness.