Microstructure evolution of AZ31 magnesium alloy during equal channel angular extrusion

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Equal Channel Angular Extrusion (ECAE), which possesses a strong grain-refinement effect through severe plastic deformation, has been employed to prepare many metallic materials with ultrafine microstructures including Al, Cu and Ti alloys [1–4]. The plasticity of magnesium alloys at room temperature is generally not good enough due to its hcp structure, thus the applications of magnesium alloys are limited. Recently, ECAE technique has been applied to magnesium alloys to improve the mechanical properties, especially the plasticity [5–7]. Mukai *et al.* found that the elongation-to-failure of AZ31 alloy, which is as high as 50% and twice that of conventionally extruded AZ31 alloy, could be greatly improved by ECAE [5]. Mabuchi *et al.* reported the low-temperature superplasticity of AZ91 processed by ECAE [6]. Kim *et al.* examined the mechanical responses and texture development of equal channel angular pressed AZ61 alloy [7]. However, researches on ECAE/magnesium alloys are mostly focussed on the relationship between microstructure characteristics and mechanical properties. The grain-refinement mechanism of ECAE/magnesium alloys has not been investigated fully up to now. Segal pointed out that the total strain intensity ε_n could be calculated by the following equation:

$$\varepsilon_n = 1.15N \cot an \frac{\phi}{2} \quad (1)$$

where N is the number of passes and ϕ is the angle between the channels [8]. Since ε_n is in direct proportion to N, the efficiency of the ECAE process is closely related to the number of passes. To answer the questions when and how the ECAE processed materials achieve their expectant refined grain structure, it is necessary to determine the microstructure evolution of ECAE process. However, little research has been conducted on this issue. In the present study, AZ31 magnesium alloy was prepared by ECAE and its microstructure after each pass was examined. Based on the experimental results, the grain refinement mechanism is discussed.

The AZ31 (Mg-A1 2.9 wt%-Zn 1.0 wt%-Mn 0.4 wt%) ingots were firstly hot extruded at 573 K into rods (22 × 22 × 120 mm) with an extrusion ratio of 4:1. The microstructure of as-extruded rods is shown in Fig. 1. ECAE was carried out in the die with an angle of 120 °C between channels. Calculated by Equation 1, the strain intensity in a single pass was 0.68. After being solution treated at 573 K for 0.5 hr, the rods were put into the ECAE die immediately and ECAE was conducted continuously through route B_c as designated in [9]. The temperature of the ECAE die was about 553–537 K. The extrusion velocity was 20 mm·s^{-1}. A mixture of graphite powder and engine oil with a volume ratio of 2:1 was used as lubricant during ECAE. No additional heating was applied to the samples during ECAE processing. Microstructures of samples processed by 2, 4, 8, and 12 passes are shown in Fig. 2.

It can be seen from Fig. 1 that the as-extruded microstructure before ECAE has an equiaxed grain structure with an average grain size of ∼25 μm. After 12 passes of ECAE processing, the microstructure was greatly refined as shown in Fig. 2d, and a homogeneous equiaxed grain structure with an average grain size of ∼3 μm was attained. It can be seen from Fig. 2 that with the increase of ECAE pass number, the grain size becomes finer. The refined equiaxed grains are evidence that dynamic recrystallization took place. However, it can be found from Fig. 2a to c that the process of grain refinement of the experimental materials is not a simple linear relationship. Fig. 2a and b shows the microstructure of 2-pass and 4-pass alloys, respectively. It is clear that the grain structures are not homogeneous but mixed structures of coarse grains and fine grains.

The main driving force of recrystallization is the stored energy of dislocations, and dislocation density increases with increasing amount of deformation. Since the experimental material is a multicrystal structure, its deformation is not uniform due to the neighboring grains needing to accomodate each other during

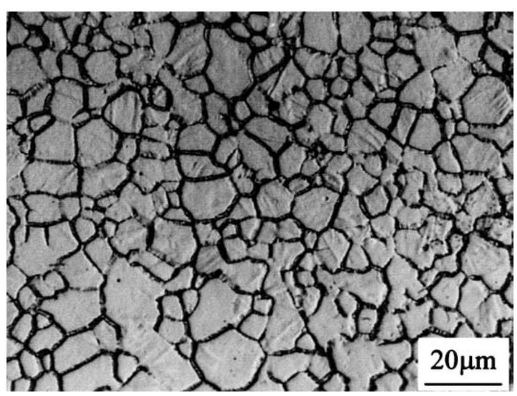

Figure 1 Microstructure of as-extruded AZ31 alloy perpendicular to extrusion direction.

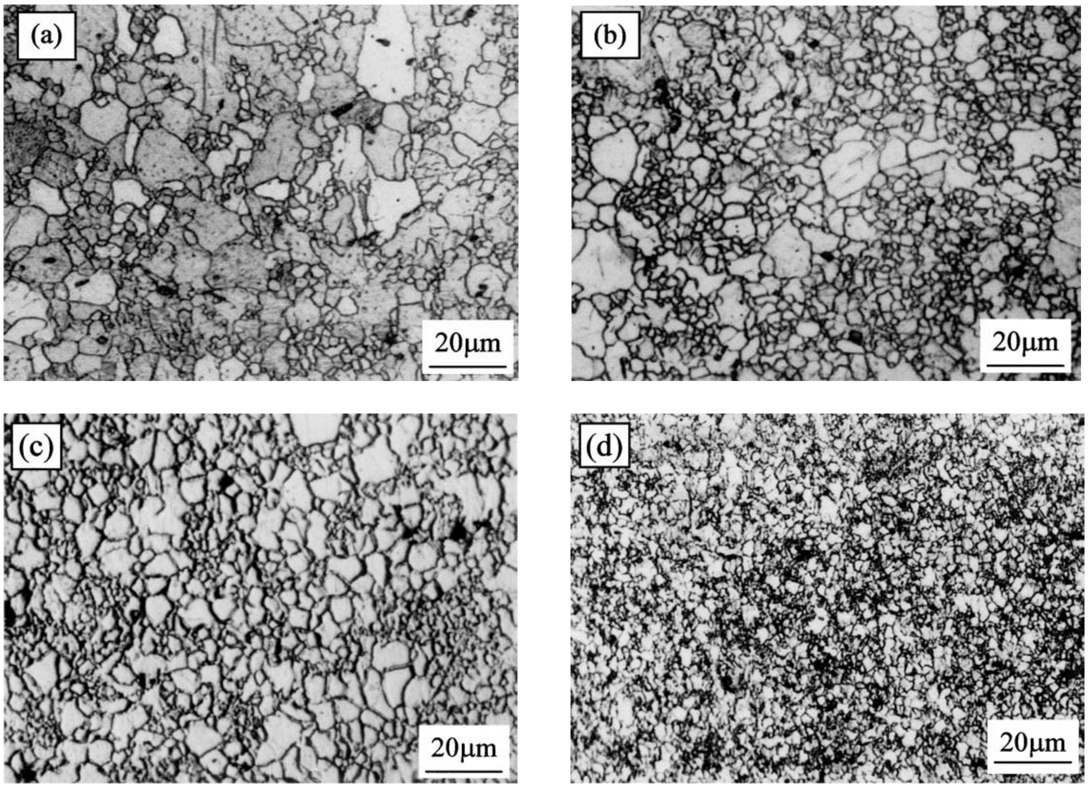

Figure 2 Microstructures of AZ31 alloys processed by ECAE with different passes: (a) 2 passes, (b) 4 passes, (c) 8 passes, and (d) 12 passes.

deforming. Dislocations generated during deformation are not distributed uniformly in the whole sample and dynamic recrystallization tends to take place at those zones where dislocation densities are high. Therefore, mixed grain structures were formed at the first stage and even during the middle stage, i.e., 4-pass processing. It is difficult to measure the average grain size in Fig. 2a and b. On the other hand, since the coarse grains and fine grains, represent the primary grains and new grains, respectively, it is meaningful to measure their size separately. In Fig. 2a, the average grain size of primary grians (D_0) and new grains (D_1) is $\sim$25 and $\sim$5 μm, respectively. It is reported that dynamic resrystallization generally begins at the primary grain boundaries, especially when $D_0/D_1 > 2$ [10]. The value of D_0/D_1 is about 5 in Fig. 2a, and many fine grains exist near the primary grain boundaries. This phenomenon is more clearly shown in the microstructure of the single pass ECAE sample, which is shown elsewhere [11]. Once dynamic recrystallization takes place and new grains are formed, dislocation density will decrease sharply at these places. In the next ECAE pass, dislocations generated by continuous deformation will accumulate in the coarse grains and consequently induce dynamic recrystallization at these places. Therefore, the fraction of fine grains increases with ECAE pass number. As shown in Fig. 2b and c, the microstructure of 4-pass and 8 pass samples remains a mixed structure while the fraction of coarse grains decreases compared with Fig. 2a. When the ECAE pass number increases to 12, the microstructure becomes uniform, and a homogenous fine grain structure with grain sizes ranging between 1 to 5 μm is achieved.

Calculated using Equation 1, the total strain intensity ε_n of 12 passes is $\sim$8. It has been found that when ECAE is carried out in a die in which the angle between the channels, ϕ, equals 90°, only eight passes are needed for AZ31 to acquire the same grain size when the other processing conditions are the same [11]. In the case of eight passes in a 90° die, the ε_n is also $\sim$8. Yamashida *et al.* prepared Mg–0.9%Al alloy by ECAE with a total strain intensity of $\sim$4 at 573 K, and the grain size was about 40 μm [12]. In Mabuchi's research, ECAE was conducted on AZ91 at 448 K with a total strain intensity of 8.05, and after annealing at 473 K for 30 min, the grain size was about 1 μm [6]. As-extruded AZ61 with an average grain size of 24.4 μm could be refined to 8.4 μm by ECAE with an equivalent strain of $\sim$8 [7]. Although the chemical composition and processing conditions are different, it may be assumed that a total strain intensity of $\sim$8 is needed for AZ series alloys to be effectively refined, especially when equiaxed grains with size less than 10 μm, which is generally one of the basic conditions for superplastic forming [13] is desired.

Besides grain refinement, microstructural changes caused by ECAE including texture transformation, grain boundary structure, remaining stress distribution, etc. also play important roles in determining mechanical properties. X-ray diffraction showed that Mg (0001) plane arrangement is quite different from that of traditional extrusion. The basal planes of magnesium, (0001) are mainly arranged parallel to the extrusion direction after traditional extrusion. As for experimental materials processed by ECAE, intensities of Mg (0001) planes are almost the same in both transverse and radial directions. Texture characteristic changes will surely influence tensile behavior, which is reported in detail in [11].

Acknowledgment
The authors would like to acknowledge the financial support from the Science Foundation of SCUT, People's Republic of China.

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Received 26 February
and accepted 6 May 2003