Processing and Mechanical Properties of Fine Grained Magnesium by Equal Channel Angular Pressing

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In this paper, grain refinement of pure magnesium using severe plastic deformation was investigated in order to enhance mechanical properties of the hard-to-deform metallic material. The microstructure and the mechanical properties of Mg processed by equal channel angular pressing (ECAP) at various processing temperatures were examined experimentally. ECAP of channel angle of 90° and corner angle of 0° was successful without fracture of the magnesium workpiece at 300°C, but not under 200°C. The hardness of the ECAP processed magnesium decreased with increasing ECAP processing temperature. The effect of temperature on the hardness, instrumented indenting response and microstructure of the ECAP processed magnesium were discussed. Fracture behavior during ECAP under different processing temperatures was demonstrated using the finite element method associated with ductile fracture model. [doi:10.2320/matertrans.MC200725]

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1. Introduction

Magnesium and its alloys are attractive as light weight structural/functional materials for high performance applications in automobile and electronics industries because of their excellent properties such as low density, high specific strength, relatively excellent dent resistance, damping capacity and shield capacity of electromagnetic waves.¹⁾ However their poor ductility due to a small number of available slip systems in the basal plane and a difficulty in dislocation activities in the non-basal plane limits further engineering applications. A general strategy to enhance ductility of metallic materials without sacrificing strength is grain refinement, which is an aim of this study.

It is generally accepted that if metallic materials are deformed at room temperature (RT), average sub-grain/cell size decreases with strain. Hence, plastic deformation processing can be a viable method for grain refinement of metallic materials. In recent years, severe plastic deformation (SPD) was developed as a new method of manufacturing bulk metallic specimens having ultrafine grained (UFG) microstructures.^{2,3)} The SPD processed materials show not only the unique physical and mechanical properties inherent in various UFG materials but also a number of advantages over nanostructured materials manufactured by other methods through powder processing. Among many SPD processes, such as accumulated roll bonding, equal channel pressing (ECAP) and high pressure torsion, the ECAP process is so far the most viable forming procedure to extrude material by using specially designed channel dies without any substantial changes in geometry and to produce UFG materials. While a significant literature on ECAP is available, most publications deal with materials having the

fcc and bcc structures. Reports on ECAP of hcp materials are rather scarce, and the processing and the deformation behavior of these materials under ECAP are not well understood. Especially, important processing variables in hard-to-deform materials, *e.g.* hcp Mg and Ti, die angles and processing temperature need to be carefully controlled to prevent fracture during ECAP.

In the present study, we employed ECAP as a method of SPD for grain refinement of pure magnesium and investigated the ECAP processing in terms of the processing temperature, the microstructural features and the mechanical properties.

2. Experimental Procedures

The material used in this investigation was commercial purity magnesium (0.11% Mn, 0.03% Si, and Mg balance). For ECAP processing, the billet was cut into 6 mm × 6 mm × 50 mm workpieces, which were surface-polished using 1200 grit SiC paper. The die parameter used, *viz.* the channel angle ($\theta_c = 90^\circ$), the outer corner angle ($\theta_o = 0^\circ$) and the inner corner angle ($\theta_i = 0^\circ$) yield a maximum effective strain of 1.155 for a single pass of ECAP. The pressing speed was 1 mm/s. A mixture of MoS₂ powder and commercial oil was used for lubrication between the workpiece and the channel surfaces.

The Vickers microhardness was measured using an Akashi HM0-122 tester by applying a load of 100 gram force for 15 s and taking an average over 7 separate measurements. Instrumented indenting behavior using a hemisphere ball indenter of 2 mm in diameter was employed.

Microstructures of the Y planes of the samples were examined before and after ECAP by optical microscopy, where the Y plane denotes the plane parallel to the longitudinal axis of the sample.

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Fig. 1 ECAP processed Mg samples. The processing temperatures are (a) room temperature, (b) 100°C, (c) 200°C, and (d) 300°C.

3. Results and Discussion

Figure 1 shows the samples after ECAP processed at RT, 100°C, 200°C and 300°C. Cracks and brittle fractures were observed for the samples which were ECAP processed at 200°C and below. The cracks were along the 45° of the sample longitudinal axis, which coincides with a shearing plane direction in the deforming zone of ECAP. The fractures occur not only from the top surfaces but also within the samples. With increasing the ECAP processing temperature, the number of cracks decreased, because the activity of non-basal slip systems,⁴⁾ hence the ductility of magnesium,

increases with increasing temperature. The activity of non-basal slip systems leading to annihilation of dislocation and deformation twinning can enhance the ductility of magnesium.⁴⁾ At 300°C, distinct cracks were not found in the sample, which is attributable to both the activities of more slip systems and the microstructural contributions, that is, the dynamic recrystallization and the dynamic recovery.

Microstructures of initial and ECAP processed pure magnesium are shown in Fig. 2. The scale bars in Fig. 2 represent 100 μm. The initial grain is equiaxed and larger than 1 mm (although micrographs on the three directional surfaces are not shown here), showing a recrystallized state

Fig. 2 Optical micrographs of (a) initial sample and (b–e) ECAP processed Mg samples. The processing temperatures are (b) room temperature, (c) 100°C, (d) 200°C and (e) 300°C.

with some twinning in the grain interior. The micrograph of the RT ECAP sample shows a severely deformed microstructure, having many shear bands and twinning. The shear banding during severe plastic deformation was well analyzed.²⁾ The former grain boundaries cannot be distinguished. Increase in the processing temperature increases internal energy and thereby promotes dynamic recrystallization (DRX), showing fewer shear bands and twins in the 200°C sample. A clearly visible microstructural feature is strong serrations of the grain boundaries, as analyzed by Estrin *et al.*⁵⁾ The formation of serrated boundaries was also observed in ECAP of AZ31⁵⁾ and compression tests performed on AZ31 at 250°C,⁶⁾ where the authors described the bulging of a grain boundary due to DRX leading to microscopic strain localization in slip lines near the grain boundaries. They assumed that the interfering dislocation slip systems on both sides of the boundary contribute to the emergence of the grain boundary serrations. This effect was

also found in other hot deformed magnesium alloy systems, such as Mg–rare earth alloys after creep tests at 300–350°C⁷⁾ and ZK60.⁸⁾ Basically, lower processing temperature ECAP is beneficial to grain refinement, hence we need to determine a minimum processing temperature where fractures are not occur.

The 300°C ECAP processed sample shows a very different and inhomogeneous microstructure, *i.e.* bimodal microstructure of several hundreds µm long band-like grains and smaller grains of ~10 µm, unlike the common equiaxed ultrafine grains in the ECAP processed Al alloys and Fe alloys. This bimodal microstructure is attributed to the recrystallization and grain growth during ECAP at temperature of higher than $1/2T_m$ ($= 188^\circ\text{C}$), where T_m is melting temperature.¹⁾ The formation of the large band-like elongated grains suggests that a possible inhomogeneous deformation could have caused local internal energy buildup and local grain growth.⁹⁾

The microhardness of the ECAP processed pure magnesium is summarized in Table 1. The hardness increased after ECAP by 9–21%, depending on the processing temperature. This increase in hardness after ECAP is attributed to the work hardening caused by the formation of subgrain bands and the increase of dislocation density occurring with the shear deformation in the initial grain interior,¹⁾ which can be found in Fig. 2, as well. The hardness decreases with increasing the ECAP temperature, because the work hardening is limited more at higher temperatures by the occurrence of the dynamics recovery, a thermally activated softening mechanism.

The hardness of 300°C ECAP processed magnesium is a little higher than the initial state due to finer grain sizes, although lower than the lower temperature ECAP processed magnesium. It is also expected that the bimodal microstructure induces strain hardening mechanism that stabilize tensile deformation, leading to enhanced elongation without sacrificing much of strength, as reported in AZ31 magnesium alloys.¹⁾ Indeed, a marked improvement in uniform elongation was found¹⁰⁾ in pure copper with bimodal microstructure consisting of micrometer-sized grains embedded inside a matrix of nano-sized grains. In addition, magnesium alloys with fine grains of less than 10 μm (this is similar to our small

grain size) introduced by ECAP activated the cross slip to not only basal plane but also non-basal plane in grain interior.¹¹⁾ Therefore, ECAP of magnesium at 300°C is best in terms of fracture and microstructural evolution under the current ECAP condition.

Figure 3 shows instrumented indentation behavior using 2 mm diameter hemispherical ball indenter. The indenting curves are consistent with the hardness testing results; high strength in 372 K sample, and decreasing strength trend with increasing ECAP processing temperature. It should be noted that the unloading gradients of all samples are the same, and elastic properties are insensitive to microstructural changes.

Theoretical simulations during plastic deformation processing are of importance in engineering processing design and optimization. The finite element/volume method is the most widely used simulation technique applied for elastic/plastic deformation and heat transfer analyses. Several researches on plastic deformation of magnesium alloys during ECAP have been analyzed using FEM/FVM.^{12–14)} However, none of the previous reports simulated fracture behavior during ECAP. Indeed, in case of ECAP for hard-to-deform materials, such as magnesium and titanium, fracture prediction as well as plastic deformation analysis is necessary. Figure 4 shows a predicted examples of the fracture simulations; ECAP of magnesium under 200°C and 300°C. The simulation results are in agreement with the experimental ones showing fracture at 200°C and sound processing at 300°C. This fracture trend with temperature can be rationalized that damage proportional to plastic deformation energy is accumulated with ECAP and ductile fracture is generated at a critical damage level. The accumulated plastic deformation energy decreases due to low stress level with

Table 1 Average hardness under different ECAP processing temperatures.

Processing Temperature	Initial	Room Temperature	100°C	200°C	300°C
Hardness, Hv	33	41	40	39	36

Fig. 3 Instrumented indentation curves of pure magnesium using a hemispherical ball of 2 mm in diameter.

Fig. 4 Simulated fracture behavior during ECAP of pure magnesium under two different processing temperatures. The numbers indicate damage levels.

increasing processing temperature. Therefore, fracture decreases with temperature. However, the critical damage level is sensitive to material, microstructure and temperature, and should be experimentally obtained. A systematic experiments

and finite element simulations for fracture behavior are underway for optimization of the ECAP processing.

4. Summary

Pure magnesium was processed by ECAP at room temperature, 100°C, 200° and 300°C. Fracture tendency decreases with increasing processing temperature, and ECAP at temperatures of 300°C and above was possible without fracture. However, grain size and inhomogeneity increase due to the dynamic recrystallization and strength decreases with increasing the processing temperature. ECAP of pure magnesium at 300°C was best in terms of fracture and microstructural evolution.

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