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# Exploration of equal channel angular pressing routes for efficiently achieving ultrafine microstructure in magnesium

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ABSTRACT

This research explored the equal channel angular pressing (ECAP) routes for efficient microstructure refinement of pure magnesium. The samples were processed for a series of numbers of ECAP passes through three different routes (i.e. A, Bc, and C routes). The utilized ECAP mold had a large channel angle of 142° and the processing temperature was 200 °C. The microstructure and texture of the processed samples were characterized using optical microscope and x-ray diffraction technique. The results show that tension twinning deformation mechanism during an ECAP pass led to the formation of profuse twins in magnesium and the twinning/detwinning dominated microstructure refinement mechanisms. After 8 ECAP passes, both Bc and C routes reduced the length and width of twins to ∼ 13 µm and ∼ 3 µm respectively. Bc and C routes can refine the microstructure of magnesium more efficiently than A route. Among the three studied routes, Bc route achieved the most homogeneous microstructure with uniformly distributed twins, while both A and C routes resulted in heterogeneous microstructure.

## 1. Introduction

Magnesium is a highly appealing type of structural metal for various structural components due to its low density, and good static and dynamic mechanical properties [1–3]. However, its absolute yield strength is relatively low. The yield strength of as-cast magnesium is reported to be as low as 20 MPa [1]. Thus, it is crucial to improve the strength of magnesium. One broadly utilized method is to add reinforcing phase [4,5] or alloying element [6]. However, this method sometimes reduces the ductility of magnesium. Another widely used method is to acquire ultrafine microstructure through a refining process and this is a proved and effective way to simultaneously enhance strength and ductility [7,8]. Microstructure refinement is usually achieved through severe plastic deformation (SPD). There are a variety of SPD processing methods such as ECAP (e.g. [9–11]), high pressure torsion (HPT) (e.g. [12–15]), accumulative roll bonding (ARB) (e.g. [16–19]), multi-pass rolling (e.g. [20–23]), and constrained groove pressing (CGP) (e.g. [24–26]). These methods were extensively employed to refine the microstructure of various metals. HPT is suitable for surface deformation and the sample can be limited to have a small size. ARB, multi-pass rolling, and CGP are ideal for processing metal sheets, while ECAP is appropriate for refining bulk metal bars.

This study focused on processing pure magnesium bars with square cross-section and ECAP was utilized to achieve ultrafine microstructure. Most reported articles on ECAP processing of pure magnesium employed a mold with the channel angle $\phi$ of 90°. For example, Kwak et al. pressed magnesium samples through an ECAP mold with $\phi = 90°$ under different processing temperatures (i.e., room temperature, 100 °C, 200 °C, and 300 °C); and they found that the samples cracked after one ECAP pass for the cases of room temperature, 100 °C, and 200 °C, while the sample did not crack when the temperature was 300 °C [27]. To avoid the cracking of samples, the ECAP temperature usually were required to be relatively high (e.g. higher than 200 °C for $\phi = 90°$). If the used temperature is low (e.g. equal to or lower than 200 °C for $\phi = 90°$), magnesium samples tend to fracture easily after one ECAP pass and could not experience multiple ECAP passes and thus could not achieve the desired microstructure refinement. Since 200 °C is ∼ 0.5 $T_{melt}$ ($T_{melt}$, the melting temperature of magnesium), the high ECAP temperature was often a barrier to the refinement of microstructure since the microstructure would experience recrystallization and grain growth during the ECAP processing. Two potential methods can be employed to improve the microstructure refinement capability of the ECAP processing. One is to apply back pressure during an ECAP processing, and the other is to use an ECAP mold with a channel angle larger than 90° (i.e. $\phi > 90°$) since this will reduce the strain experienced by the sample per ECAP pass. Jäger and Gärtnerová utilized back

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pressure during an ECAP processing in a mold with 90° channel angle [28]. With the employment of back pressure, ECAP could be performed at room temperature and four ECAP passes were applied to magnesium with the back pressure increased from ~ 200 MPa for the first pass to ~ 400 MPa for the fourth pass [28]. Poggiali et al. processed magnesium by three ECAP passes through the route Bc and the mold had a channel angle of 135° [29]. Jiao and Li utilized an ECAP mold with a channel angle of 142° to process pure magnesium for 8 passes through the route Bc at 200 °C [11]. Although most of the reported research efforts employed the ECAP mold with $\phi = 90°$, the ECAP mold with $\phi > 90°$ was also used for various metals other than magnesium. Nakashima et al. performed the ECAP processing of pure aluminum using the molds with the channel angles of 90°, 112.5°, 135°, and 157.5° respectively [30]. Gholinia et al. utilized an ECAP mold with the channel angle of 120° to refine the microstructure of Al–3% Mg–0.2% Zr–0.2Fe and Al–0.3% Mn [31]. Liu et al. studied the microstructure and mechanical properties of Cu–38% Zn alloy experienced cumulative large plastic deformation using an ECAP mold with the channel angle of 160° [32]. Dheda et al. studied the effect of initial microstructure on the ECAP processing of titanium using a mold with the channel angle of 120° [33].

This study chose to follow the large channel angle method. Although some reported articles used one route (i.e. the Bc route) for ECAP processing through a mold with large channel angle (e.g. [11,29]), there are multiple different ECAP routes for processing a sample. Among these routes, three widely-utilized ones are: A route, the billet is not rotated; Bc route, the billet is rotated 90° clockwise or counter-clockwise; and C route, the billet is rotated 180° [34]. In this study, these three different ECAP routes (A, Bc, and C) were utilized in the processing of pure magnesium through a mold with $\phi = 142°$ and the number of ECAP passes was up to eight for each ECAP route to explore the effectiveness of various ECAP routes on refining microstructure. The microstructural information of samples experienced the different types of ECAP routes will facilitate the design of processing routes for achieving different microstructure features and guide our selection of the processing parameters to achieve certain microstructure feature efficiently.

## 2. Experimental

This work employed ECAP technique to process commercially pure magnesium with a purity of 99.9%. The samples for ECAP processing were prepared to be in a cubic shape and have a dimension of ~ 10 mm × 10 mm × 35 mm. Before ECAP processing, all samples were heat treated at 300 °C for 12 h. As shown in Fig. 1, the 'L' shaped ECAP mold had a channel angle $\phi$ of 142° between the entrance channel and the exit channel and a curvature angle $\psi$ of 0° at the connection of the two channels. The cross section of the entrance channel is the same as that of the exit channel, and these cross sections are the squares of 10.5 mm × 10.5 mm. Fig. 1 also sketched the extrusion direction (ED), the transverse direction (TD), and the normal direction (ND). The approximate strain $\varepsilon_N$ can be computed for a sample experienced $N_{ECAP}$ ECAP passes using the following Eq. (1) [35].

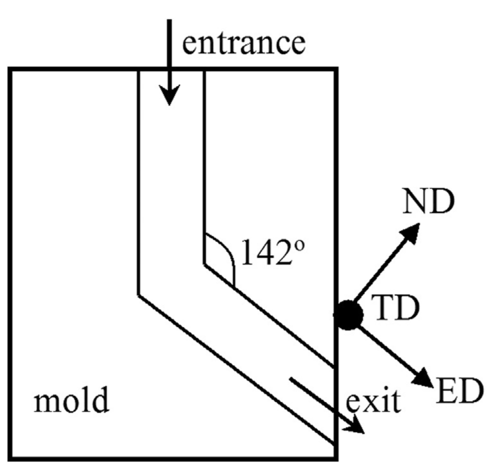


**Fig. 1.** The geometry of an ECAP mold with a channel angle $\phi$ of 142° between the entrance channel and the exit channel. ED, ND, and TD are extrusion direction, normal direction, and transverse direction respectively.

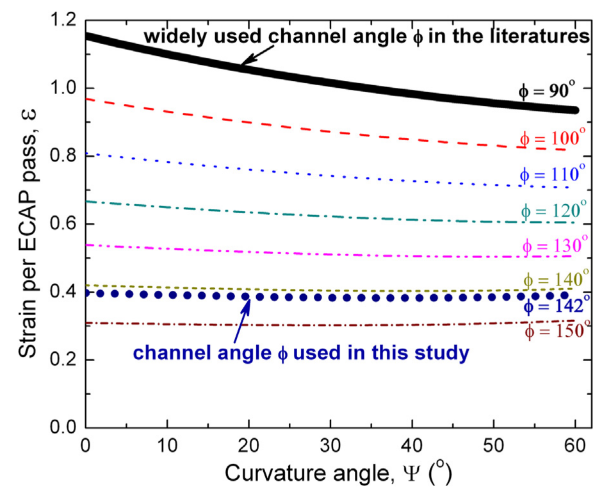


**Fig. 2.** The map of strain per ECAP pass when the channel angle $\phi$ varies in the range of 90–150° and the curvature angle $\psi$ varies in the range of 0–60°. $\phi = 90°$ is the most widely-used channel angle in the published literature, and $\phi = 142°$ is the channel angle used in this study.

$$\varepsilon_N = \frac{N_{ECAP}}{\sqrt{3}}\left[2\cot\left(\frac{\phi+\Psi}{2}\right) + \Psi\mathrm{cosec}\left(\frac{\phi+\Psi}{2}\right)\right] \tag{1}$$

During each ECAP pass, the strain will be estimated by setting $N_{ECAP}$ to be 1 in Eq. (1). Fig. 2 presents a map of the strain per ECAP pass with respect to the curvature angle $\psi$ in the range of 0–60°, while fixing the channel angle $\phi$ at a series of values (i.e. 90°, 100°, 110°, 120°, 130°, 140°, 142°, and 150°). When $\phi$ is in the range of 90–140°, the increase of $\psi$ reduces the strain per pass. The increase of $\phi$ leads to a decreasing strain reduction rate with the increase of $\psi$. For the angle 142° used in this study, there is a negligible change in the strain when the angle $\psi$ changes. The strain per pass for $\phi = 142°$ is about 0.4. Although the compressive facture strain of magnesium is usually less than 0.2 at room temperature, it can bear the strain of 0.4 at 200 °C without cracking [36]. The utilization of $\phi = 142°$ for the mold allows magnesium samples to go through the mold without being fractured at 200 °C and thus to be able to experience more than one repeating ECAP pass.

Table 1 lists the ECAP processing parameters in this study. For each ECAP pass, the temperature of mold ($T_m$) was 50 °C and the temperature of sample was 200 °C. Between two consecutive ECAP passes, a sample was heated at 200 °C for 10 min. Three sets of samples were processed through ECAP. *Set 1* followed the A route and there was no sample rotation between two consecutive ECAP passes, and the samples were processed for 1, 2, 4, and 8 passes and they were designated as A1, A2, A3, and A4 respectively. *Set 2* followed the $B_c$ route and there was 90° sample rotation between two consecutive ECAP passes, and the samples were processed for 1, 2, 4, and 8 passes and they were designated as A1, B2, B3, and B4 respectively. *Set 3* followed the C route and there was 180° sample rotation between two consecutive ECAP passes, and the samples were processed for 1, 2, 4, and 8 passes and they were designated as A1, C2, C3, and C4 respectively. The ECAPed samples were ground, polished, and then etched using an acetic-picral etchant for microstructure observations. The etchant was prepared according to the following ratio: 5 ml acetic acid, 6 g picric acid, 10 ml water, and 100 ml ethanol. The samples were also studied using the x-ray