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Original Article

Effect of grain size on compression behavior of magnesium processed by Equal Channel Angular Pressing

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A B S T R A C T

Commercially pure magnesium was processed by a single pass of Equal Channel Angular Pressing (ECAP) at 523 K. The grain structure was evaluated at the region near the intersection between the channels of the die. Compression test workpieces were machined from the as-received material and the material processed by ECAP. These workpieces were tested at room temperature. The results show the grain structure is refined in the intersection between the channels. The ECAP processed material displays higher strength and reduced surface bulging due to the refined structure. The plastic flow during compression of the ECAP processing material concentrates along bands of refined grains.

1. Introduction

There is an increasing interest on the mechanical behavior of magnesium and its alloys due to the potential use of these materials in transportation industry in place of heavier materials. Early papers reported the mechanical behavior of magnesium alloys tested in compression at high temperatures [1,2]. It was shown that dynamic recrystallization takes place at temperatures above ~423 K and the new grains nucleate along grain boundaries of the old grains. Recent papers have shown that this mechanism of dynamic recrystallization takes place during Equal Channel Angular Pressing (ECAP) leading to pronounced grain refinement in magnesium and its alloys [3,4]. ECAP [5] is a severe plastic deformation technique in which a billet is pressed through a die with two channels of equal cross section that intersect at an abrupt angle. The billet is sheared during its passage through the intersection between the channels without any change in its cross-section. Therefore, the process can be repeated imposing large amounts of strain to the material.

ECAP has been extensively used to process magnesium and its alloys leading to significant grain refinement and improved mechanical properties. For example, an elongation to failure in tension of up to ~40% was reported in a magnesium alloy processed by ECAP [6]. Later it was shown that this increase in ductility is observed only in certain loading directions [7].

The behavior in tension of magnesium alloys processed by ECAP is now well known. However, there are only few papers

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Fig. 1 – Appearance of the longitudinal section of samples of magnesium alloy after compression at different temperatures [1].

describing the behavior in compression. It is known that magnesium alloys exhibit flow localization during compression at high temperatures [1]. Fig. 1 shows the appearance of samples of extruded magnesium alloy tested in compression at temperatures in the range 423-593 K. It is observed that the longitudinal section of the samples attain distorted appearances due to the occurrence of flow localization. The distortion of the sample becomes more pronounced at the lower temperatures. However, testing was not carried out at room temperature. Moreover, these samples were processed by extrusion. It is of interest to study the behavior at room temperature of samples processed by ECAP. A recent paper reported successful processing of pure magnesium by ECAP at 523 K and refinement of the grain structure to produce an average grain size of 24 μm [8]. Thus, the present paper aims to describe the evolution of the grain structure of pure magnesium during processing by ECAP and the behavior of this material during room temperature compression.

2. Experimental material and procedures

The material used in this work is commercially pure magnesium provided by Rima as as-cast slabs. Cylindrical billets with 9.8 mm diameter and 60 mm long were machined from the as-received material and processed by ECAP. The ECAP die had angle between channels of 90° and an external curvature in the area of intersection between channels characterized by an angle of 45°. Processing was carried out at 523 K with punch speed of 0.1 mm/s. A lubricant with molybdenum disulfide was used to reduce friction between the billets and the die walls. An earlier paper [8] showed the punch load during ECAP is ~1 kN and there is significant contact friction between the billet and wall despite the use of lubricant. Pressing was interrupted before the whole billet had crossed the intersection between channels. A sample was cut out of the billet in the region near the deformation zone in order to evaluate the grain structure. The sample was mounted, grinded, polished and etched to reveal the grain structure.

Cylindrical samples with 4 mm diameter and 6 mm height were machined out of the as-received material and the material processed by ECAP for compression tests. The axial direction of the samples was parallel to the axial direction of the billet after ECAP. Compression tests were carried out in an Instron universal testing machine model 5582. Molybdenum disulfide based lubricant was used between the samples and the compression plates in order to reduce friction effects during the test. The tests were carried out at constant rate of cross-head displacement with initial strain-rate of 10^{-3} s^{-1}. Load data was recorded during the test by a load cell with 100 kN capacity. The distance between ink marks in the sample was tracked by a video device attached to the testing machine and converted into strain. True stress and true strain curves were calculated considering homogeneous deformation of the sample.

The compression tests were interrupted after significant drop in load which was caused by fracture of the sample. The samples were photographed using magnifying lenses after testing and then mounted using resin. The mounted samples were ground in abrasive papers #180, #400, #600 and #1000 in order to remove a surface layer of material and observe the longitudinal section of the sample. Final polishing was carried out using 1 μm alumina suspension and then the sample surface was etched with a solution of 5% nitric acid in ethanol to reveal the grain structure. Images of the sample longitudinal section were recorded using optical microscopy.

3. Experimental results

Fig. 2 shows a montage of images of the grain structure in the area near the intersection between the channels. The inlet channel is vertical and the outlet channel horizontal. The material on the top left has not crossed the shear zone and the initial coarse grain structure is observed. Many twins are observed in this area which suggests that some deformation takes place before the material crosses the shearing zone. The material on the bottom right has crossed the shearing zone and the grain structure is significantly refined.

The stress vs. strain curves for the as-received material and the material processed by ECAP are shown in Fig. 3. The as-received material is depicted in a solid line while the material processed by ECAP is depicted in dashed line. The as-received material exhibits a low yield stress of ~20 MPa and pronounced strain-hardening. A peak stress of ~110 MPa was observed at a strain of ~0.13 and a drop in stress was

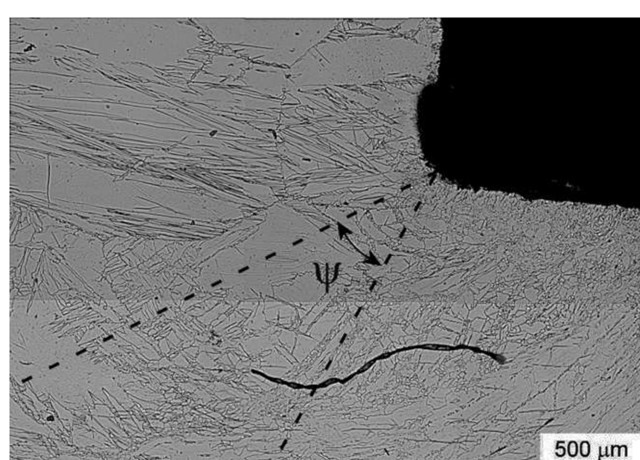

Fig. 2 – Grain structure in the region near the intersection between channels of the ECAP die. The dashed lines delineate the theoretical shearing zone.

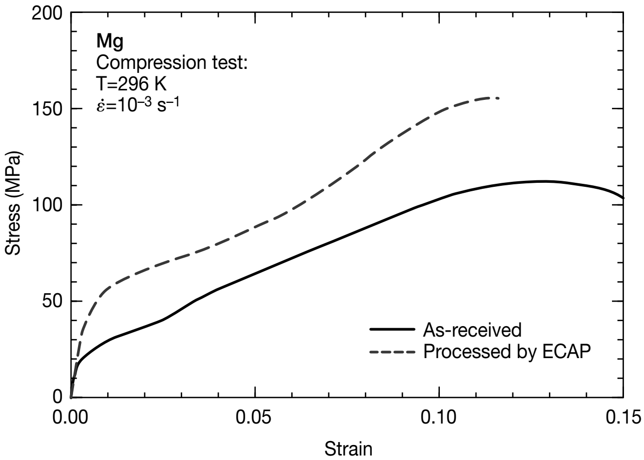

Fig. 3 – Stress vs. strain curves for the as-received material and the material processed by ECAP.

observed thereafter. This apparent softening is attributed to the formation of a fracture in the sample.

The ECAP processed material displays higher yield stress of ~50 MPa than the as-received material but a similar strain hardening. A peak stress was observed at ~150 MPa at a strain of ~0.11 followed by an apparent softening caused by fracture of the sample.

Fig. 4 shows the appearance of the samples after being tested to failure in compression. It is observed that both samples exhibit fracture in regions with apparent concentration of shear. This suggests that significant amount of deformation took place in the region before fracture. The as-received material exhibits the formation of coarse bumps on the surface. These bumps have dimensions of > 1 mm which is similar to the dimensions of the average grain size. They are attributed to inhomogeneous deformation of neighboring grains. These bumps are not observed in the ECAP processed material which has significantly finer grains [8].

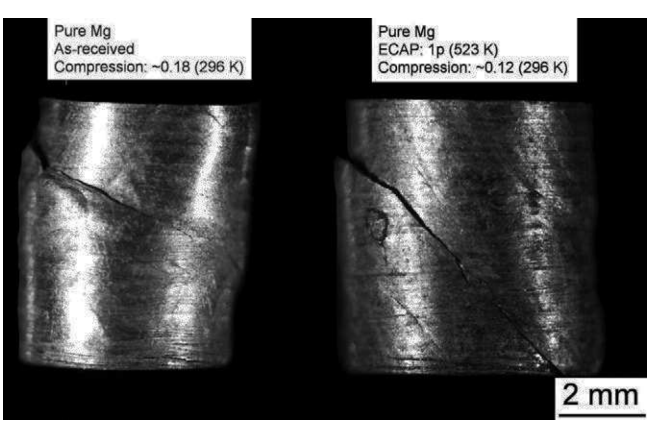

Fig. 4 – Appearance of the workpieces of the (left) as-received material and (right) material processed by ECAP after being tested in compression at room temperature up to failure.

The polished and etched longitudinal sections of the compression samples are shown in Fig. 5. The as-received material is shown on the left and the ECAP processed material on the right. The as-received material exhibits coarse grains of ~1000 μm and it is observed that the fracture initiates in the region between two coarse grains. A few intragranular shear bands were observed.

Fig. 6 shows images of pure magnesium processed by ECAP and subjected to compression along the axial direction. The loading direction is the vertical direction in the images. The grain structure of the ECAP processed material is heterogeneous. Some coarse grains of over 100 μm are visible but they are surrounded by fine grains of ~10 μm. Alternating bands of coarse and refined grains are clearly observed away from the fracture in Fig. 6a and the fracture along a band of refined grains is observed in Fig. 6b. It is apparent that bands of fine grains are formed at a direction of ~45° to the sample axial direction. Therefore the bands of fine grains seem to be formed parallel to the shearing plane of ECAP. Moreover, it is observed that shear deformation tends to take place parallel to these bands. In fact, the fracture took place in a region of finer grains.

4. Discussion

4.1. Grain refinement

Early papers discussed the mechanism of grain refinement in magnesium and its alloys processed by ECAP [1,2]. It was shown that grain refinement is caused by dynamic recrystallization

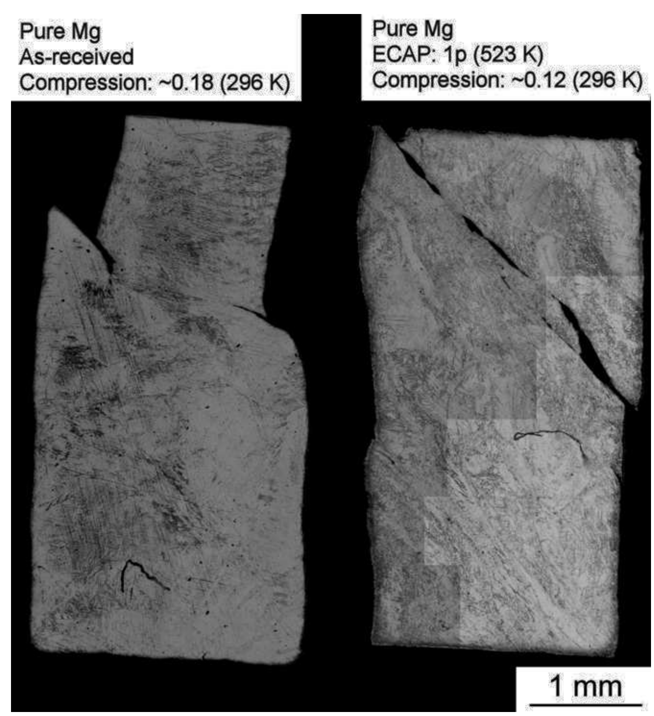

Fig. 5 – Grain structure along the longitudinal section of the workpieces of the (left) as-received material and (right) material processed by ECAP after being tested in compression at room temperature up to failure.

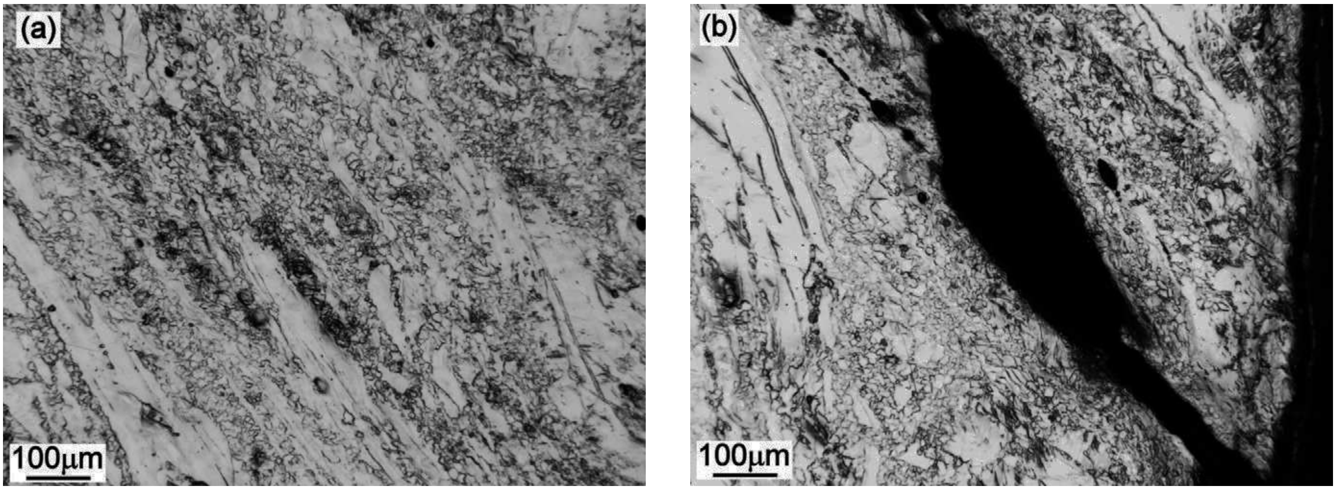

Fig. 6 – Grain structure of pure magnesium processed by ECAP and subjected to compression along the axial direction (vertical direction in the images). Alternating bands of refined grains and coarse grains (a) away from the fracture and (b) fracture along bands of refined grains.

and the new grains are formed along original grain boundaries leading to a bi-modal grain size distribution. This observation is in agreement with the model of dynamic recrystallization in magnesium alloys proposed in earlier papers [1,2]. A recent paper [8] showed the bi-modal grain size distribution is also observed in pure magnesium provided the processing temperature is low in order to prevent significant grain growth of the new grains.

The present paper confirms the formation of the bimodal grain size distribution in pure magnesium and it shows that the new grains form preferentially along bands of fine grains oriented parallel to the ECAP shearing plane. This is clearly observed in the longitudinal section of the compression sample of material processed by ECAP where layers of coarse grains and layers of fine grains are inclined at a direction of ~45° to the sample axial direction. This is the same orientation of the shearing plane in ECAP during prior processing.

The formation of bands of recrystallized grains was observed by Ion et al. [1] in a magnesium alloy. The authors proposed a mechanism of formation of shearing bands which is shown in Fig. 7. The new grains are originally formed along grain boundaries in a necklace pattern. The new grains are favorably oriented for shearing and they will accommodate most of the ongoing plastic deformation. Therefore, continuation of deformation leads to broadening of these layers until the formation of a continuous thick shearing band that crosses several grains.

The amount of shear strain imposed by ECAP is believed to be sufficient for the formation and growth of these shearing bands. Therefore the layers of fine grains observed in the longitudinal section of the compression sample of the material processed by ECAP are in agreement with the model of shearing bands in magnesium.

4.2. Mechanical behavior

The present results show that the as-received material exhibits significant bulging of the surface (a coarse 'orange peel' effect)

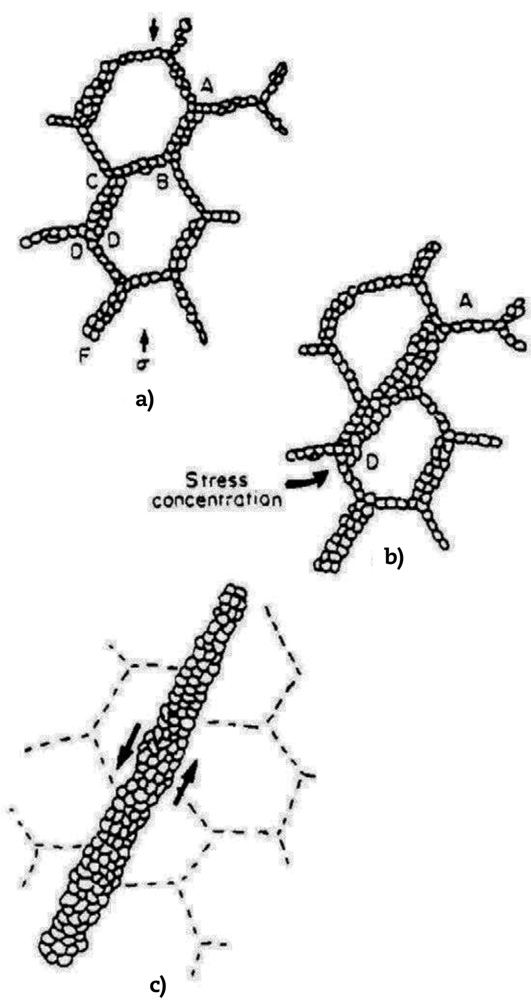

Fig. 7 – Illustration of the mechanism of formation of shearing bands during dynamic recrystallization of magnesium alloy proposed elsewhere [1].

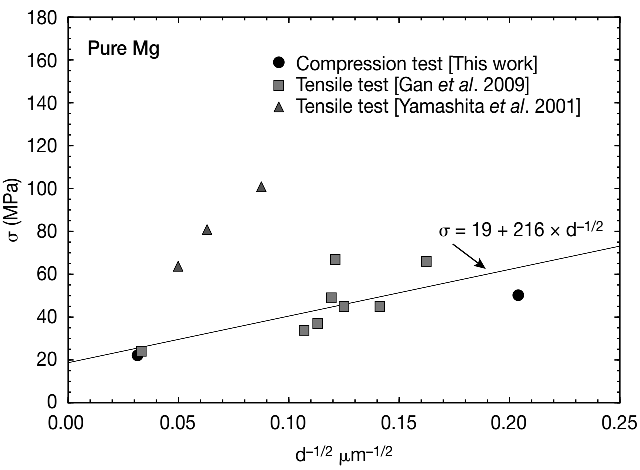

Fig. 8 – Flow stress as a function of the inverse of the square root of the grain size for pure magnesium processed by ECAP.

due to the coarse grain structure. This might prevent the use of these materials for forming of components with small dimensions. However, processing by a single pass of ECAP leads to pronounced grain refinement and bulging of the surface is not visible.

The grain refinement leads also to an increase in flow stress as expected. However, this increase is not very high. The initial flow stress, σ, of the material processed by ECAP, ~50 MPa, is lower than that reported during tensile testing of coarser grained pure magnesium processed by ECAP [9,10]. In order to facilitate the comparison of the present results with others reported in the literature, the initial flow stress, σ, of pure magnesium is plotted as a function of the inverse of the square root of the grain size in Fig. 8. It is worth noting that the flow stress was determined by compression tests in the present paper and tensile testing in the others. It is observed that the data do not follow a single trend. For example, the flow stress reported by Yamashita et al. [9] is significantly higher than that observed in the present paper despite the finer grain structure in the latter. This shows that structural characteristics other than the grain size play a key role on the mechanical behavior of magnesium.

A trend line was drawn through the data of the present results and the data reported by Gan et al. [10]. This trend line suggest a flow stress for a single crystal of pure magnesium of ~19 MPa and a Hall-Petch constant of 216 MPa $\mu m^{1/2}$. Further testing is required in order to determine the relationship between flow stress and grain size in pure magnesium processed by ECAP.

4.3. *Texture*

Although texture was not determined in the present study its effect should be commented. It is expected that the as-received material does not exhibit any preferential orientation of its structure because it was provided as as-cast slabs. However, Ion et al. [1] report that the grains formed by dynamic recrystallization exhibit a preferential orientation in which its basal plane is aligned to the shearing plane. This preferential orientation is retained throughout the deformation which suggests a continuous dynamic recrystallization.

The shearing plane in ECAP is oriented at 45° to the billet axial direction. Therefore it is expected that the new grains will attain a preferential orientation in which the basal planes are oriented ~45° to the axial direction. In fact, the formation of texture in pure magnesium was observed by Gan et al. [10] after one pass of ECAP. Thus, the ECAP processed material would exhibit not only a bimodal grain size distribution but also a bimodal texture. The coarse grains could retain the random orientation while the fine grains would exhibit preferential orientation. Moreover, the fine grains would be favorably oriented for shearing during compression along the billet axial direction. This is in agreement with the observed shearing lines in the samples of ECAP processed magnesium alloys which are parallel to the bands of fine grains.

5. Summary and conclusions

- Pure magnesium was processed by one pass of ECAP from the as-cast condition. The grain structure and the mechanical properties were evaluated.
- Bands of fine grains are formed inclined to the axial direction of the billet during processing by ECAP. Shearing concentrates along these bands during subsequent compression along the billet axial direction.
- Pronounced bulging (a coarse 'orange peel' effect) is observed on the sample surface during compression of the as-cast material. This is attributed to the coarse grain structure. ECAP processing refines the grain structure and prevents such surface effects.
- The observed slip lines in the sample processed by ECAP and subjected to compression are in agreement with a mechanism of formation of fine grains favorably oriented for slip along the shearing plane at which dynamic recrystallization occurred.

Acknowledgements

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