INVESTIGATION OF MECHANICAL PROPERTIES OF ECAPED AZ31 MAGNESIUM ALLOY WITH RARE-EARTH ELEMENTS AND Ca ADDITIONS BY SHEAR PUNCH TEST

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Abstract

Microstructural refinement is an effective way for increasing both ductility and strength of poor formable Magnesium alloys. This goal can be achieved by a variety of techniques including processing by equal channel angular pressing (ECAP). In the present study, microstructure and mechanical properties of ECAPed AZ31 magnesium alloy with 0.6%RE, 0.6%Ca and 0.3%RE-0.3%Ca additions, were investigated. After extruding, the materials were ECAPed for 4 passes using route BC. The effect of equal channel angular pressing on the room temperature mechanical behavior of experimental alloy was studied using shear punch testing (SPT) method. Because of textural effect induced by ECAP, AZ31 showed lower yield stress and higher ductility in comparison to the as-extruded condition, in spite of strengthening effects due to grain refinement. Addition of the RE elements and calcium increased tensile strength mainly by dispersive strengthening effects of particles, and improved tensile ductility of the extruded material by texture modification. The introduction of RE elements and calcium in the AZ31 also resulted in the refinement of the grain structure, mainly because of the suppression of the dynamic recrystallization by RE and Ca-containing particles during hot deformation process.

Keywords: AZ31; RE elements; Calcium; ECAP; Texture; Mechanical properties

1. INTRODUCTION

Magnesium alloys are becoming increasingly attractive for engineering applications because of their particularly low density, excellent damping capacity, good recycling capacity and machinability [1]. However, Mg alloys have poor formability and limited ductility at room temperature due to their hexagonal closed packed (hcp) structure with limited slip systems. Accordingly, many attempts have been made to improve the formability of wrought Mg alloys [1,2]. Microstructural refinement is an effective way for increasing both ductility and strength of these alloys. This goal can be achieved by a variety of techniques including processing by equal channel angular pressing (ECAP) [3-5]. In this process, the cast or extruded material is pressed in an L-shaped die to undergo a very high shear strain deformation without any change in the cross-sectional area. In most investigations, because of the low ductility of Mg alloys, they are ECAPed at or above 200 °C to avoid cracking.

Mechanical properties of Mg alloys strongly depend on crystallographic texture, mainly due to the limited slip systems of their hcp structure at room temperature. Despite the grain refinement achieved by ECAP of Mg alloys, their yield stress often decreases while ductility is improved. This behavior has been attributed to an unusual texture, where the basal planes are highly inclined (~45°) to the extrusion axis [6-9]. There have been some attempts to prevent softening of Mg alloys processed by ECAP. Kim et al. [9] employed direct extrusion and rolling and achieved significant increase in strength, although tensile ductility decreased. Practical way to enhance mechanical behavior of Mg alloys has been concerned through using alloying elements. Among these elements, rare earth (RE) and calcium additions are of special interest due to their impact on the casting characteristics, high temperature mechanical properties and corrosion resistance [10-13]. Wu et al. [13] showed...
that adding 1.05% Ce to AZ31 alloy increases the strength and tensile ductility after rolling and annealing. Liu et al. [14,15] reported that extruded AZ31 alloys with 0.3 and 0.6% RE additions had the best mechanical properties, but 1%RE addition decreased the ductility because of the coarse Al11RE3 precipitates formed at grain boundaries. They also showed that the AZ31 Mg alloy sheets with 0.3%RE and 0.3%Ca additions had the best bending and drawing–bulging formability. Jun et al. [16] studied the effects of Ca additions on the mechanical properties of an Mg-RE-Zn cast alloy. They showed that with increasing Ca content, yield stress increased gradually, whereas elongation showed a decreasing trend.

It is well documented that the tensile strength data can be related to the effective shear strength data obtained through the shear punch testing (SPT) method of thin specimens for a variety of materials [17,18]. This permits a means of evaluating the flow properties, when only a small amount of material is available, particularly in the ECAPed samples with limited dimensions. The load–displacement curve obtained from the punch shearing operation exhibits many similarities to that of tensile test curves, e.g., initial linear elastic region, yield point, a plastic deformation region and an ultimate load. Guduru and his co-workers [19] have thoroughly studied the effect of sample thickness, die–punch clearance and strength of materials on the variation of yield and ultimate strength measured by SPT. They reported a good correlation between the shear punch and tensile data for yield and ultimate strengths.

In the present work, microstructure and mechanical properties of ECAPed AZ31 magnesium alloy with 0.6%RE, 0.6%Ca and 0.3%RE-0.3%Ca additions, were investigated. The effect of equal channel angular pressing on the room temperature mechanical behavior of experimental alloy was studied using shear punch testing (SPT) method.

2. EXPERIMENTAL PROCEDURE

Four alloys which were designed for this investigation have the nominal chemical compositions of Mg-3 wt.% Al-1.0 wt.% Zn-0.3 wt.% Mn (AZ31), AZ31-0.6Ca, AZ31-0.6RE and AZ31-0.3Ca-0.3RE. High purity Mg (99.90 wt.%), Al (99.84 wt.%), Zn (99.99 wt.%) and Ca (99.9 wt.%) were used to prepare the alloys. Melting was carried out in an electrical furnace held at 750 °C under the Foseco MAGREX 36 covering flux to protect molten magnesium form oxidation. Lanthanum-rich misch-metal (RE) together with other elements were added to the melt and held for 30 min to make sure that they were completely dissolved. Melts were then poured in a preheated steel die by a tilt casting technique in order to minimize casting defects and turbulences of the melt. Cast billets of 44 mm diameter were homogenized at 385°C for 12 h and then extruded to 11×11 mm bars at 370°C. The ECAPed billets having dimensions of 10×10×80 mm were machined from the extruded bars. ECAP was performed at 200°C through the die with channel angles Φ=90° and ψ=20°. The samples subjected to repetitive pressings were rotated by 90° in the same sense between each pass in the procedure designated route Bc. This configuration leads to an imposed strain of 1 on each passage through the die. Samples were sprayed with MoS2 lubricant and pressed at a speed of 1 mm/s for 4 passes.

Microstructure of the side faces (i.e. Y plane [9]) of the extruded and ECAPed billets at the exit from the die was studied by optical metallography and scanning electron microscopy (SEM). The metallographic samples were polished using 0.03 μm α-Al2O3 and then etched using a solution of 4.2 g picric acid, 10 ml acetic acid, 10 ml distilled H2O and 70 ml ethanol. The intensity distribution of the {0002} pole figures representing the basal plane
was measured by the Schultz reflection method of the Y plane of ECAPed specimens. The measurement was performed using Cu kα radiation at 50 kV with the sample tilt angle ranging from 0° to 90°.

Shear punch tests were performed using a screw driven MTS material testing system with a load cell of 20 kN capacity and a crosshead speed of 0.10 mm min⁻¹. Some 0.8-mm thick slices were cut from the ECAPed bars perpendicular to the ED. These slices were carefully ground to a thickness of 500 μm and located in a specially designed fixture with a 3.175-mm diameter flat cylindrical punch and 3.225-mm diameter receiving-hole. After application of the load, the applied load P was measured automatically as a function of punch displacement; the data were acquired by a computer so as to determine the shear stress of the tested materials using the relationship

$$\tau = \frac{P}{\pi dt}$$

where P is the punch load, t is the specimen thickness and d is the average of the punch and die diameters. Three different samples were tested for each condition and the variation in the measured ultimate shear strength values was small.

3. RESULTS AND DISCUSSION

Figure 1 shows the SEM microstructure of the as-cast AZ31, AZ31-0.6RE, AZ31-0.6Ca and AZ31-0.3RE-0.3Ca alloys. It can be observed that the microstructure of AZ31 (Fig. 1a) consists of α-Mg matrix and β-Mg₁₇Al₁₂ particles. After the addition of 0.6% RE elements, some needle-shaped Al₁₁RE₃ particles appear in the as-cast microstructure, as shown in Fig. 1b. At the initial stages of the solidification of the RE-containing alloys, RE elements have a strong affinity to combine with aluminum. Consequently, with the precipitation of the plate-like Al₁₁RE₃ phase, β- Mg₁₇Al₁₂ disappears. When La- and Ce-rich mischmetals are added to the Mg-Al alloys, the intermetallic particles are either Al₁₁Ce₃ or Al₁₁La₃. Due to the similar effects of these two elements, however, they are generally known as Al₁₁RE₃. The precipitates in the microstructure of AZ31-0.6Ca alloy, shown in Fig. 1c, have the same chemical composition of β-Mg₁₇Al₁₂ phase with some dissolved calcium. It is believed that this dissolution enhances the thermal stability of β-Mg₁₇Al₁₂ at elevated temperatures [20]. In the AZ31-0.3RE-0.3Ca (Fig. 1d), both β-Mg₁₇Al₁₂ and Al₁₁RE₃ particles are present.

The microstructures of 4 passes ECAPed AZ31, AZ31-0.6RE, AZ31-0.6Ca, and AZ31-0.3RE-0.3Ca alloys are shown in Fig. 2. The grain structure of AZ31 (Fig. 2a) has become reasonably uniform with an average grain size of about 1.6 μm. In the alloys containing RE elements and Ca, however, the fragmented particles are dispersed in the matrix after ECAP.

The equiaxed grains in microstructures imply that dynamic recrystallization has occurred during the ECAP process. The fragmented particles in the grain boundaries of RE- and Ca-containing alloys suppress the dynamic recrystallization and increase the nuclei for recrystallization. There is also ample heat to initiate grain growth during cool-down because the dynamically recrystallized microstructure is not quenched after extrusion and ECAP. Therefore, the intermetallic particles may have a retardation effect on the moving grain boundaries during grain growth. This has resulted in a finer grain structure that is achieved in the RE- and Ca-containing alloys which the AZ31-0.3RE-0.3Ca alloy has the lowest grain size of 1.2 μm.
Fig. 1. SEM micrographs of the as-cast alloys (a) AZ31; (b) AZ31-0.6%RE; (c) AZ31-0.6%Ca; (d) AZ31-0.3%RE-0.3%Ca.

The (0002) pole figures of the AZ31 and AZ31-0.3RE-0.3Ca alloys in the extruded and after being ECAPed for 4 passes are shown in Figure 3. In the as-extruded alloys (Figs. 3a and c), it is evident that (0002) basal planes are mostly oriented parallel to the extrusion direction. After 4 passes, however, (0002) basal planes are inclined about 45° to the extrusion axis, as shown in Figs. 3b and d. The comparison of (0002) pole figures of the AZ31-0.3RE-0.3Ca alloy with that of AZ31 shows that the precipitates have not affected the final positions of the texture. However, the intensity of the maximum orientation in both of the extruded and ECAPed materials is decreased by RE and calcium additions. This decrease is about 50% for the extruded materials and about 25% for the ECAPed conditions. This drop might be due to the randomly particle-stimulated nucleation of recrystallization that results in different orientations in the recrystallized microstructure and to the lower grain boundary mobility during grain growth caused by the presence of intermetallic particles [21]. The smaller drop in the intensities of the ECAPed materials in comparison with the extruded conditions can be due to the higher strains in the ECAPed conditions that facilitate the nucleation of recrystallization, irrespective of the particles presence.

Figure 4 compares the shear punch behavior of the AZ31, AZ31-0.6RE, AZ31-0.6Ca and AZ31-0.3RE-0.3Ca alloys after 4 ECAP passes with that of the extruded AZ31 alloy. There are two important findings. First, yield shear stress decreases after 4 ECAP passes, though the grain size decreases considerably. Secondly, the shear ductility increases with increasing the number of passes. Furthermore, after 4 passes of ECAP, alloys have approximately the same ultimate shear strength values as those of the extruded materials, although their yield shear stresses are lower. This may imply that the strain hardening capacity of all materials has increased
after 4 ECAP passes. It can also be observed that with adding RE elements and calcium, the strength of the 4-passed AZ31 alloy increases but its ductility decreases. In comparison with the extruded AZ31 alloy, the 4-passed AZ31-0.6RE, AZ31-0.6Ca and AZ31-0.3RE-0.3Ca alloys have higher shear strength and ductility.

![Fig. 2. SEM micrographs showing the microstructure after 4-passes ECAP (a) AZ31; (b) AZ31-0.6%RE; (c) AZ31-0.6%Ca; (d) AZ31-0.3% RE-0.3%Ca.](image)

The shear punch behavior of the extruded and ECAPed materials, shown in Fig. 4, indicates that the yield shear stress of the ECAPed AZ31 alloy is lower than that of the extruded alloy, despite more effective grain refinement caused by the ECAP process. This may be attributed to the texture modification occurring in the hcp crystal structure of Mg during ECAP. Primary slip occurs on the (0001) basal planes in Mg alloys at room temperature because of their low critical resolved shear stress, as compared to those of the non-basal slip systems on prismatic and pyramidal planes [22]. For the extruded alloys, the basal planes tend to lie parallel to the extrusion direction (Figs. 3a and c), implying that the primary slip on the basal plane would be difficult, and thus the strength is improved. However, the Schmid factor on the (0001) basal planes increases by the rotation of the basal planes (~45°) during the ECAP process (Figs. 3b and d), and thus a lower stress is needed for yielding of the ECAPed materials.

Another feature of the ECAPed materials is their enhanced ductility respect to the extruded conditions. Similar to the strength properties, grain refinement and texture modifications are the main reasons for the observed ductility enhancements. However, the grain-size dependence of tensile elongation is not sufficient to explain the large increase of the tensile ductility. Mukai et al. [6] applied the ECAP technique to AZ31 alloy to reduce the grain size to 1 μm. After annealing which was accompanied by substantial grain growth, the alloy retained its relatively high tensile elongations at room temperature. This was then attributed to the texture modification.
However, this large increase of the tensile ductility cannot be achieved only by the activation of the basal planes due to texture modifications. This is because the basal slip provides only two independent slip systems, far fewer than the necessary five independent systems for homogeneous deformation, according to the Von-Mises criterion [23]. Consequently, some prismatic and pyramidal slip planes have also been activated easily because of a rotation of about 45° from the extrusion axis by ECAP. The significant strain hardening observed in the shear punch testing curves of the ECAPed materials is in agreement with the argument that two or more slip systems are active during tensile testing.

Fig. 3. (0002) pole figures of AZ31; (a) extruded, (b) 4-passes ECAPed, and for AZ31-0.3% RE-0.3%Ca (c) extruded, (d) 4-passes ECAPed.

The effect of the alloying elements on the tensile deformation behavior of the ECAPed materials is shown in Fig. 4. Similar to the extruded conditions, the addition of the RE elements and calcium increases the shear strength and decreases the ductility. The interesting point is that in most of the ECAPed alloys the ductility is higher than that of the extruded AZ31 base alloy. This can be attributed to the texture modification which occurs during ECAP. Concerning the strength properties, the ECAPed RE- and Ca-containing alloys have approximately the same strength as the extruded AZ31 alloy, despite the occurrence of texture softening due to the ECAP process.
Fig. 4. Shear punch test curves of 4-passes ECAPed AZ31, AZ31-0.6%RE, AZ31-0.6%Ca and AZ31-0.3%RE-0.3%Ca alloys with extruded AZ31 alloy.

4. CONCLUSIONS

In this investigation, the effects of the rare earth elements and calcium additions on the microstructure and mechanical properties of AZ31 magnesium alloy are studied by shear punch testing method, in both extruded and ECAPed conditions. The introduction of RE elements and calcium in the AZ31 resulted in the refinement of the grain structure, mainly due to the suppression of the dynamic recrystallization by RE and Ca-containing particles during the hot deformation process. Furthermore, the added alloying elements did not change the final position of the maximum orientations in the pole figures, but decreased their intensities. From the point of view of mechanical properties, in comparison to the extruded materials, the ECAPed alloys showed lower yield shear stress and higher shear ductility, because of the texture modification by the ECAP process. The RE- and Ca-containing ECAPed AZ31 alloy can benefit from high strength caused by the dispersive strengthening effects of the particles, and high ductility imparted by texture modifications.

ACKNOWLEDGEMENTS

The authors wish to thank the Research Council of Islamic Azad University-Sirjan Branch for providing financial support of this work.

LITERATURE


