Microstructures and Mechanical Properties of Microalloyed and Equal Channel angular Pressed AZ91 Mg Alloy

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Abstract. Commercial AZ91 Mg alloys were equal channel angular pressed (ECAP) for up to 6 passes at temperature as low as 473K. Microstructures and mechanical properties of as-received and as-pressed samples were investigated. It is shown that ECAP applied successfully to these materials at elevated temperatures leads to grain refinement due to the occurrence of recrystallization during the pressing process and to significant improvements in the strength and ductility of these materials. The microstructure was obviously refined and the average grain size was considerably reduced from over 28μm to below 1μm. From tensile testing at room temperature (RT), the ultimate tensile strength (UTS) increases obviously from 318MPa to 365MPa, in contrast, the uniform elongation increased from 12% of the as-received to 26% of the pressed up to 4 passes. These observations may be attributed to the effects combined grain refinement and texture development.

1. Introduction
Magnesium alloys are attractive for light-mass structural applications in the transportation and aerospace industry, which seeks to reduce weight and increase specific strength [1]. However, most Mg alloys exhibit severe limitations in their ductility, strength and creep resistance due to a hexagonal close-packed crystal structure. The easy slip systems in the hexagonal close-packed materials such as pure Mg and Mg based alloy are very limited, compared with those in cubic materials. A large number of grains are initially in the so-called hard orientations with small Schmid factors where deformation is difficult to occur.

These inherent difficulties may be reasonably overcome by grain refinement under some special processing [2-6].Equal channel angular pressing (ECAP) can provide a promising technique for gaining microstructures with ultra-fine grains in size range from the sub-micrometer to nanometer in bulk materials by introducing extremely large plastic straining during deformation processing, and has been proven to be very effective processing in improving the mechanical properties [2]. There are many experimental evidences that great grain refinement may be achieved using ECAP, and there are advantages because it can give large bulk samples and can scale up for commercial applications by using multi-pass facilities [7].
In the present research, equal channel angular pressing (ECAP) was applied to a commercial AZ91 magnesium alloy for up to 6 passes at temperatures as low as 473K. Microstructures and mechanical properties of as-received and as-pressed samples were investigated for better understanding of the effect mechanism of multiple-pass pressing with related to the number of ECAP pass.

2. Experimental procedures
The detailed chemical compositions (mass fraction, %) of AZ91 the commercial magnesium alloy used in present study are: 8.6 Al, 0.8 Zn, 0.25 Mn, 0.004 Fe, 0.003 Cu, 0.001 Ni, and balance Mg. Rod samples of AZ91 magnesium alloy were cut in diameters of 16 mm and lengths of 100 mm. The ECAP was conducted at 473 K at a pressing speed of ~15 mm s\(^{-1}\) and a die with an angle of 90° of the two channels and an external angle of 20° of the outer arc of curvature where the two channels intersect. So that these two channels give an imposed shear strain of ~1 on each pass of the sample through the die. Each rod was heated in the die at the selected temperature for ~20 min prior to the first pass and then held at the same temperature again for ~10 min prior to each subsequent pass. Repetitive pressings were conducted at that temperature with the specimens rotated around their axes by 90° in the same direction and coated with molybdenum disulphide (MoS\(_2\)) as a lubricant between each pass in route Bc. The temperature of the process was controlled through a thermocouple fixed in the die.

Experimental study of microstructure was carried under optical microscopy (OM). For OM, samples were cut parallel to the extrusion direction and carefully polished to a mirror plane and then etched in a solution of 4.2 g picric acid, 10 ml acetic acid, 10 ml H\(_2\)O and 70 ml ethanol. Tensile specimens with gauge length of 30 mm and diameter of 5 mm were machined from the as-received sample and also from the rods produced by ECAP, of which the gage length was aligned along the longitudinal direction of the pressed rods. The tests of tension were carried out by an Instron1185 machine at room temperature with a rate of 0.5 mm/min.

3. Results and discussion
Microstructures. Fig.1 shows the OM images of as-received and those ECAP processed specimens, and the mean grain diameter was determined in linear intercept method. The grain size of the original microstructure of the as-received is typically nonuniform on the cross sections, and the mean diameter was about 28\(\mu\)m as show in Fig.1 (a). The microstructures after ECAP at 473 K are shown in Fig. 1 (b-f). Although many grains were already significantly refined after only 3 passes, the grain structure was not homogeneous with very fine grains of 0.5–3\(\mu\)m as well as few grains of 5\(\mu\)m. And those coarse grains being surrounded by fine ones were also observed by other investigators [8-11]. Some grains are refined with little residual strain whereas other large grains undergo distortion with traces of plastic deformation. The decomposition of the grain-structure attributing to ECAP began in the grain boundary area, in another word the dynamic recrystallization began [12], as it can be seen in Fig.2 (b). However, it resulted in the bimodal microstructure that the dynamic recrystallization was not complete. With further ECAP processing at the same temperature, the volume fraction of fine grains increases while that of large grains decreases. A homogeneous fine microstructure with an average grain size of 1\(\mu\)m after 5 passes of ECAP can be obtained. The average grain size is further homogeneous after 6 passes of ECAP. It was possible that grains with more favorable orientations were deformed and refined first, leaving islands of less deformed and coarser grains, since the slip systems in Mg were very limited. Eventually, deformation spread to all the grains and the microstructure became homogeneous [12].

In the present experimental work it shows that the grains were reasonably equiaxed and homogeneously distributed for further ECAP pressing at the temperature as low as 473K. This homogeneity after only four passes suggests that recrystallization occurs during ECAP, and the occurrence of recrystallization is consistent also with the advent of larger grain sizes in the Mg alloy by comparison with pure Al [9] and commercial Al-based alloys [11].
**Tensile properties.** Fig. 2 shows the tensile properties of the as-received and those ECAP processed specimens. It can be seen from Fig. 2 (a) that, the ultimate tensile strength (UTS) of the alloy increases rapidly after one pass, then slowly and steadily increases up to six passes, but decreases slightly after 5 passes. The plasticity increased significantly up to 4 passes before decreasing slightly up to 5 passes.

Figure. 1 Optical photographs of the microstructure of (a) as-received, (b) one pass, (c) three passes, (d) four passes, (e) five passes, (f) six passes.

The tensile results showed that the strength of the materials ECAP deformed at 473K initially increased from 318MPa to 365MPa and the ductility increased significantly from 12% in the extruded...
material to 26% after 4 passes before decreasing. These properties are similar to those observed in former studies [13-14]. Other factors than grain sizes should be considered besides the grain size decreased monotonously with the number of passes. As is known to all that Mg alloys would develop a fiber texture after extruding with \(<10\overline{1}0>\) in the basal plane aligned with the extrusion direction. As the basal planes would be parallel to the tensile direction, this is a hard orientation. This texture would gradually decrease with ECAP deformation and be replaced by another one with the basal planes parallel to the shear plane in ECAP deformation which is at 45° to the longitudinal direction [8]. The gain in strength due to grain refinement is believed to be more than the loss of strength due to texture softening, through comprehensive consideration the strength was improved after ECAP.

The 45° texture of Mg alloy constitutes a soft orientation with lower strength and higher ductility [8]. On the contrary with AZ61[14], at the beginning of tensile deformation, the grain refining was more significant, resulting in strength increase; later, the texture softening effect arrived at an equilibrium to the grain refining stabilised with the completion of one cycle of the Bc route ECAP deformation [8]. Not the same as the strength, the ductility increased quickly owing to combination between the grain refinement and texture softening in the uniform elongation after 1 ECAP pass. The subsequent decrease in uniform elongation might be attributed to a decrease in strain hardening rate as the new texture became well established in most grains during further ECAP.

![Graph](image_url)

**Figure. 2** Dependence of (a) ultimate tensile strength and (b) elongation on the number of ECAP passes

4. Conclusion

(1) AZ91 magnesium alloy was successfully ECAP deformed for up to 6 passes at temperature as low as 473K and the average grain size was considerably reduced from over 28μm to below 1μm.

(2) The microstructure was initially not uniform with a “bimodal” grain size distribution after one pass but became increasingly homogeneous with further ECAP passes.

(3) The UTS increases clearly after one pass, then increases continuously and slowly after the following 2-6 passes. Not the same, the uniform elongation increased significantly up to 4 passes, then followed by slowly decrease up to 6 passes. These observations may be attributed to combined effects of grain refinement and texture development.

Acknowledgements

This work was financially supported by the natural science foundation of Fujian Province (2014J01168) of China.

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