Microstructure and mechanical properties of pure magnesium prepared by CEE-AEC at different temperatures

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Abstract

Cyclic expansion extrusion with an asymmetrical extrusion cavity (CEE-AEC) was carried out on pure magnesium up to 3 passes at different deformation temperatures of 250 °C and 350 °C. The microstructure and texture evolution of its edge and center regions are studied respectively, and their mechanical properties are correlated. The results show that there is an incomplete dynamic recrystallization (DRX) region in pure magnesium deformed at 250 °C. Therefore, pure magnesium processing at 250 °C has a larger grain size and higher texture strength than that processing at 350 °C. According to the tensile test, the ultimate tensile strength (UTS) of different positions of different temperatures is very close because the DRXed grains grow at 350 °C. But the value of tensile yield strength (TYS) is nearly doubled, the main reasons are that the effect of (0001) basal slip and texture softening is greater than that of grain refinement.

1. Introduction

The density of magnesium is 1.73 g cm\(^{-3}\), which is about two-thirds of aluminum and one-fourth of steel. As the lightest metal structure material, it has great application potential in weight reduction usages [1–3]. However, its strength and plastic properties at room temperature are limited by its hexagonal close-packed (HCP) structure, and affect its development in practical applications [4, 5]. To date, many studies have been conducted on the methods of improving the mechanical properties of magnesium and its alloys, and it has been found that obtaining ultra-fine grains by severe plastic deformation (SPD) is an effective way [6–8].

The researches on SPD method have aroused widespread attention, such as high-pressure torsion (HPT) [9–11], equal channel angular pressing (ECAP) et al [12–14], multi-directional forging (MDF) [15, 16] and cyclic expansion extrusion (CEE) [17–21]. We can find from the above several SPDs, like ECAP and HPT, that high shear strain is the key to producing ultra-fine grains (UFG) and influence the grain orientation [22, 23]. CEE is a relatively new SPD method, which is characterized by simple dies and a combination of tensile and compressive stresses during deformation. On the basis of CEE, we added shear stress by using an asymmetrical extrusion cavity, and did not change the advantages. We called this modified deformation method as cyclic expansion extrusion with an asymmetrical extrusion cavity (CEE-AEC). Yan et al [24] found Mg-Gd-Y-Zn-Zr obtained UFG after CEE-AEC processing, and the DRXed grains have different effects on the texture as the passes increase. Zhen et al [25] studied the microstructure and texture of AZ31 prepared by CEE-AEC at different temperatures. However, the information about the microstructure, texture, and mechanical properties at different regions of the pure magnesium processed by CEE-AEC at different temperatures is still unknown.

In this article, pure magnesium was subjected to CEE-AEC at temperatures of 250 °C and 350 °C respectively. The effect of temperature on the texture and grain size of pure magnesium in the CEE-AEC process was studied, and its influence on mechanical properties was explored. This research provides basic process parameters for CEE-AEC.
2. Experimental procedures

2.1. Principles of the CEE-AEC

Figure 1 (a) shows the process of CEE-AEC. There are 2 steps in the process. Firstly, put the cushion block into the bottom of the cavity and put the billet on the cushion block. The billet is filled in the cavity with the punch pressed down. Secondly, remove the cushion block and put the second billet in the cavity. As the second billet fills the cavity, the first billet is extruded from the bottom of the die to complete the first pass of deformation. When performing the second pass, only the steps in the red dashed box in figure 1 are required. It should be noted that the deformed billet should be rotated 180° along the extrusion direction before putting it in.

2.2. Materials and steps

The material used in this paper was 50 mm × 100 mm × 250 mm pure magnesium processed from cast ingots. The material is homogenized at 300 °C for 16h before extrusion. Put the billet into the CEE-AEC die with 50 Rockwell hardness tool steel as the main part and fixed by annealing screw, seen in figure 1 (a). The die is characterized by asymmetrical left and right cavity, which introduces shear stress according to the different flow of the block blank. To reduce the friction between the die and the blank, the oil-based graphite is used as a lubricant during the extrusion process, and extrusion on the 6300 KN hydraulic press at a speed of 1 mm s⁻¹. CEE-AEC was carried out at 250 °C and 350 °C to 3 passes. Both the die and the samples are heated to an adapted temperature and kept for 1h before extrusion, and its microstructure, texture, and mechanical properties are observed. In order to study the effect of CEE-AEC on the microstructure and mechanical properties of the core and edge region of the CEE-AEC sample, the microstructure observation and tensile test samples were taken from two representative locations in the sample as shown in figure 1 (b): both samples are from the middle area parallel to the ED direction of the specimen, the difference is that one is in the edge area (A) and another is in the center area (B).

The processed tensile specimens were tested for mechanical properties along the ED direction on the 3382 Instron universal material experiment machine at room temperature. With an initial strain rate of 0.001 mm s⁻¹, the mechanical properties including ultimate tensile strength (UTS), yield strength (YS), and elongation (EL) are tested. The EBSD sample was electropolished in a mixture of 10ml perchloric acid and 90 ml alcohol at minus 30 °C, and the texture evolution was studied by electron backscatter diffraction (EBSD) on Hitachi SU5000 scanning electron microscope (SEM) at a voltage of 20 kV, the inclination angle of 70°, and the working distance of 15 mm.

![Figure 1](image-url)
3. Results and discussion

3.1. Microstructure

The microstructure of homogenized pure magnesium processed by CEE-AEC is characterized by EBSD band contrast as shown in Figure 2. And the observation direction of EBSD inspection is parallel to the extrusion direction. White lines are low-angle grain boundaries (LAGBs), while black lines are high-angle grain boundaries (HAGBs). As can be seen in Figure 2, the grain size distribution is inhomogeneous, and there are small grains around larger grains, which is due to DRX. At the same time, the number of large grains at the edge and center of 250 °C is significantly more than 350 °C. Generally, the strain distribution of the edge region and the center region of the sample is different, the edge region accumulates larger stress, and the grains are much finer. However, as seen in figures 3(a), (b), the average grain size in the edge region of the sample at 250 °C is 31 μm, which is larger than the average grain size of 26 μm in the central region. It may be due to the presence of the incompletely DRX region of the sample at 250 °C, as marked by the blue circle in Figure 2(a) which is different from the fine grains region in the blue box in Figure 2(d), resulting in inhomogeneous grain size distribution, and the larger average grain size in some regions. When at 350 °C, the edge region grains are significantly finer, and the number of large grains in the center region is less than that at 250 °C, as shown in figure 3(c), (d), this may be caused by the growth of the DRXed grains, furthermore, the maximum grain size of 93 μm in figure 3(d) also confirmed the growth of DRXed grains. It can be inferred that the grain size of pure magnesium prepared by CEE-AEC at 350 °C is finer than that at 250 °C. Because at this temperature, the effect of DRX on grain refinement is greater than the effect of grain growth.

Figure 4 shows the misorientation angle distribution at different positions of pure magnesium prepared by CEE-AEC at different temperatures. Sub-grains with a misorientation of 2°~15° are LAGBs, and those with a misorientation of >15° are high-angle boundaries. Lei et al [26] have proposed that low-angle grain boundaries decrease, and high-angle grain boundaries increase, indicating that recrystallized grains increase to form a large number of equiaxed grains. It can be seen that the pure magnesium processed by CEE-AEC at 350 °C has obviously more HAGBs (figure 4), indicating that there are more dynamic recrystallized grains in this state. It also shows that DRX at this temperature is more sufficient.

Figure 2. Microstructures of pure Mg characterized by EBSD band contrasts (a) edge region at 250 °C, (b) center region at 250 °C, (c) edge region at 350 °C, (d) center region at 350 °C, blue circle is incompletely DRX region, blue box is fine grain region.
Figure 3. Grain size distribution of pure Mg (a) edge region at 250 °C, (b) center region at 250 °C, (c) edge region at 350 °C, (d) center region at 350 °C.

Figure 4. Misorientation distribution of pure Mg (a) edge region at 250 °C, (b) center region at 250 °C, (c) edge region at 350 °C, (d) center region at 350 °C.
3.2. Texture evolution

Figure 5 gives the corresponding EBSD-based (0001) pole Figures on the ED-TD plane at the center and the edge of 250 °C and 350 °C, respectively. It can be seen that with the temperature increasing, the texture intensities are significantly weakened, the strength of the edges decreased from 18.9 to 12.1, and the center decreased from 27.9 to 18.8. Zhang et al found that the coarse grains can lead to a strong basal texture, but the basal poles in the regions of DRX grains present a rather dispersive distribution [27]. It also proves that the preparation of pure magnesium at 350 °C is easier to produce DRX grains than at 250 °C. Moreover, the texture is inclined along the extrusion direction (ED) and parallel to the transverse direction, which may be related to the introduction of shear strain in the CEE-AEC asymmetric cavity. As shown in figures 5(b), (d), the texture of the center is similar at 250 °C and 350 °C, and another weaker pole intensity point was developed. Meanwhile, at the two temperatures, the grain size in the middle of the sample is shown in figures 3(b) and (d). The texture intensity at 350 °C is 18.8, which is lower than that at 250 °C. Therefore, it can be concluded that texture will affect its mechanical properties, which will be discussed in the next section.

The Schmid factor of basal, prismatic, and pyramidal on the edge of samples processed by CEE-AEC at the temperature of 250 °C and 350 °C is shown in figure 6. The average SF of the (0001) (11-20) basal surface slip of the edge region of the 250 °C sample is 0.33. It can be seen that the value of SF tends to improve with the increase of temperature. Therefore, the average SF value of the edge region of the 350 °C sample increased to 0.35. And the evolution law of the average SF value of the prismatic slip system and the pyramidal slip system is consistent with that of the basal slip system. It is well known that the TYS of the sample is related to the critical resolved
shear stress (CRSS) required for the activation of the slip system. In addition, the increase of the average SF value will reduce the CRSS of the corresponding slip system [28]. Therefore, the sample proceeded at 350 °C is more conducive to dislocation slipping on the planes than that proceeded at 250 °C.

3.3. Mechanical properties

Figure 7 displays the tensile stress-strain curves of the edge and center regions of pure magnesium processed by CEE-AEC at 250 °C and 350 °C, respectively. Table 1 shows the mechanical properties at the edge and center regions of pure Mg alloy after 3 passes processed at 250 °C and 350 °C. It can be clearly seen that the TYS and the UTS of the sample at the edge and the center which processed at 250 °C are both higher than the sample processed at 350 °C, and the EL rate is opposite, showing that the mechanical properties at the edge region are superior to that at the center region when processed at 250 °C and 350 °C. It is worth noting that as the temperature increases from 250 °C to 350 °C, the UTS of the edge and the center do not change much, but the YTS decreases from 49MPa, 46MPa to 25MPa, 23MPa, respectively. The reason for this significant difference is that the YTS is not only affected by the average grain size, but also related to the effect of texture on the mechanical properties and the activation of the slip system.

As shown in figure 2, the number of large grains of the sample processed at 350 °C is less than that processed at 250 °C, and the distribution of grains is more homogeneous. It indicates that 350 °C is more conducive to DRX than 250 °C in the CEE-AEC. However, the average grain size of the center at different temperatures is 26
(figures 3(b), (d)), this is because the DRXed grains grow at 350 °C, which is also the reason why the UTS of CEE-AEC samples at different temperature is not much different. Xu et al [29] studied the TYS of Mg alloys at room temperature was mainly determined by the operation of basal dislocation slip. The sample processed at 350 °C has a higher Schmid factor than 250 °C (figure 6), so dislocations are more likely to slip on planes of the sample processed at 350 °C. As widely known that texture also has an impact on mechanical properties [30]. A weaker texture will weaken its mechanical properties. It can be obtained from figure 5 that the texture strength of both the edge and the center is significantly reduced as the temperature increases from 250 °C to 350 °C, and the values have been reduced from 18.9 and 27.9 to 12.1 and 18.8, respectively. Meanwhile, the YTS of the edge and center decreases from 49MPa and 46MPa to 25MPa, 23MPa as the processing temperature increased. The improvement of the mechanical properties of the sample by grain refinement is weakened, and the effect of (0001) basal slip and texture softening is greater than that of grain refinement.

4. Conclusions

In this paper, we studied the effect of CEE-AEC forging pure magnesium at 250 °C and 350 °C on the microstructure evolution and mechanical properties of its edges and center, and obtained the following conclusions:

1. The pure magnesium processed by CEE-AEC has formed dynamically recrystallized grains at 250 °C and 350 °C, and grains are refined. However, there is an incomplete DRX area at 250 °C, and the grain distribution is inhomogeneous. As a result, the pure magnesium prepared by CEE-AEC at 350 °C will produce more dynamic recrystallized grains than 250 °C.

2. Upon the CEE-AEC process, the grain size at the edge is much smaller than that at the center due to the inhomogeneous stress distribution. After dynamic recrystallization, the DRXed grains grow up at 350 °C. In addition, the samples at the center have the similar pole figures and average grain size at different processing temperatures.
3. Pure magnesium processed at different temperatures has close UTS, and the YTS processed at 250 °C is significantly higher than 350 °C. This performance indicates that the grain size dominates the UTS, while the YTS is mainly affected by the Activation of basal slip and the texture softening.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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