Effect of Multi-Pass Equal Channel Angular Pressing on the Microstructure and Mechanical Properties of a Directional Solidification Mg$_{98.5}$Zn$_{0.5}$Y$_{1}$ Alloy

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The microstructure evolutions and mechanical properties of a directional-solidification (DS) Mg$_{98.5}$Zn$_{0.5}$Y$_{1}$ alloy during multi-pass equal channel angular pressing (ECAP) were systematically investigated in this work. The results showed that there was a large amount of lamellar structure in DS alloy. These lamellar structure have almost the same orientation inside the grains. After ECAP, dynamic recrystallization channel angular pressing (ECAP) were systematically investigated in this work. The results showed that there was a large amount of lamellar structures have almost the same orientation inside the grains. After ECAP, dynamic recrystallization channel angular pressing (ECAP) were systematically investigated in this work. The results showed that there was a large amount of lamellar structures have almost the same orientation inside the grains.

1. Introduction

Mg–Zn–Y alloy have been regarded as the most promising high-strength magnesium alloy due to its high room temperature strength, high temperature resistance, good electromagnetic shielding performance and high creep resistance. Long-period ordered phase (LPSO), as a common second phase in Mg–Zn–Y alloys, has high temperature stability and good deformation ability. What needs special attention is that the interface of LPSO with the magnesium matrix is well-coordinated. This feature can accommodate a certain degree of deformation. Last but not least, the LPSO play an important role on the corrosion. Damping performance and hydrogen storage performance of the alloy. The above advantages of LPSO phase attracted a lot of research attention. Recently, much of the work about LPSO has focused on the regulation of its morphology. The strengthening effects of LPSO phases with different morphologies are different. For example, the diffusely distributed fibrous LPSO phase can effectively increase the strength of the alloy, and the larger volume of LPSO phase will lead to the insufficient dispersion and grain refinement. Therefore, how to change the morphology and distribution of LPSO phase is a hot topic in recent years.

In the research of changing the morphology of LPSO phase, there are mainly two ways, controlling the solidification process and plastic deformation. As early as 2001, Kawamura and Inoue studied rapidly solidified Mg$_{57}$Zn$_{1}$Y$_{2}$ alloy and suggested that the formation area of the LPSO phase was not fixed. In the initial stage, the formation of the LPSO phase were usually at the grain boundary. Then, as the temperature increased during subsequent processing, the LPSO phase also appeared inner the grains. Meanwhile, in the rapidly solidified Mg$_{50}$Zn$_{24.6}$Y$_{13.6}$ alloy, the decrease of component segregation played an important role in the change of LPSO phase morphology. Except the rapid solidification, directional solidification is also commonly used to change the morphology of the LPSO phase. Muto studied the directional solidified Mg–Y–Zn alloy, the result showed that a large number of lamellar structures appeared in the alloy, and the direction of these lamellar structure were parallel to the direction of heat flow. In terms of plastic deformation, the most common approaches include reciprocating extrusion, ECAP, etc. Lu studied the Mg$_{97.1}$Zn$_{0.8}$Y$_{0.1}$ prepared by multi-pass ECAP at 375°C, the results suggested that after 16 passes, the grain size decreased to 1 µm. Meanwhile, a large number of LPSO phases fractured, the yield strength, tensile strength and elongation of the alloy reached 387 MPa, 324 MPa, 23.2%, respectively. The improvement of mechanical properties was ascribed to refined grain and diffused distribution of small LPSO phases. Many studies have focused on the deformation of LPSO. Their results showed that a series of deformations (bending, kink, and fracture) occurred in the LPSO phase. Combined the good coherent with the Mg matrix, the LPSO phase can effectively accommodate deformation. Such results show that the control of the solidification and plastic deformation are effective ways to change the morphology of the alloy. Although extensive research about directional solidification and ECAP have been carried out, the combination of both is barely found. How does the directional solidified affect the subsequent ECAP? The effect of combination on the morphology of grain is not clearly clarified.

Based on the above background, in this study, the Mg$_{98.5}$Zn$_{0.5}$Y$_{1}$ alloy were prepared by directional solidification and fabricated by multi-pass ECAP. The microstructure evolutions and mechanical properties were systematically investigated. In particularly, the deformation mechanism of LPSO phase and the effect of LPSO on dynamic recrystallization was discussed.

2. Experiment Procedures

The alloys with the nominal compositions were prepared by induction melting pure Mg (> 99.8%), pure Zn
(> 99.8%), Mg-25%Y (mass%) in the boron nitride (BN) coated graphite under a dynamic argon gas atmosphere. To prepare DS polycrystal whose crystal growth directions were oriented along the solidification direction, the ingots were directionally solidified using the power down method with a cooling rate of 1°C/min under an Ar atmosphere. The samples for ECAP processing were taken from the DS ingot with the dimensions of 12×12×50 mm³. Each pass of ECAP was carried out using a special die with die angle of 120°. The samples were subjected to 1, 2 and 3 passes of ECAP at 573 K, 673 K. Then the compress tests were performed. The microstructure and crystal structure were analyzed with a transmission electron microscope. Phase identification were performed by X-ray diffraction (XRD, Rigaku D/max-2500PC) using a copper target with a scanning angle from 10° to 90° and a scanning speed of 2° min. The bright-field TEM images were obtained from a JEOL JEM 2100F FEGTEM instrument operating at 200 Kv. Thin foils for TEM observation were prepared by cutting the bulk sample into slices, grinding to the thickness of about 50 µm, and ion milling finally.

3. Results and Discussion

Figure 1(a) and (b) show the OM image and EBSD results obtained from the as-DS Mg₉₈.₅Zn₀.₅Y₁ alloy. As seen in the Fig. 1(a), the crystal growth direction in the DS alloy exhibited a peculiar regularity. There are a great number of lamellar structure within the grains (Fig. 1(b)), which is the LPSO phase. As shown in the EBSD images (Fig. 1(c) (d)), the grain size is 600 µm on average, and the growth direction of grain is almost the same.

For further analysis of the LPSO phase, the microstructure was clarified by the bright-field TEM image in Fig. 2. Figure 2(a) and (d) are quoted from our previous work.¹⁵) These lamellar structure have a different width, as shown in Fig. 2(c). Figure 2(d) shows that the [1030] selected-area electron diffraction (SAED) patterns of the LPSO in the as-DS-grown state, thirteen diffraction spots are observed in the (a *) reciprocal-lattice row. The diffraction spots are distributed evenly between the incident beam and the (0002) fundamental reflection of the hcp unit cell. These diffraction spots indicate that the LPSO phases in these crystals exhibit the 14H structure.

Figure 3 shows the microstructures obtained from the Mg₉₈.₅Zn₀.₅Y₁ alloy after different numbers of ECAP passes. The ED and the LD are indicated by the arrows. From the picture, we can tell that after 1-pass ECAP, the grains are dramatically refined. In contrast to the as-DS condition, the size of grains decreased to 20~30 µm. After 2-pass and 3-pass extrusion, the grain size is 10 µm on average, and the effect of grain refinement is far less than the first extrusion. The material is subjected to shear stress in the equal channel, which changes the arrangement of the LPSO phase. During the deformation process, the LPSO phase is bent and the twin crystal is formed at the grain boundary. Large amount of DRXed Mg grains can be observed along grain boundaries and LPSO phase boundaries. As the temperature
risected, the fraction of the DRXed region increased. From the Fig. 3, we can conclude that the increase in temperature is conducive to the occurrence of dynamic recrystallization. The possible reason for such a phenomenon is the dynamic recrystallization that occurs with the thermal deformation process. The driving force is the difference in dislocation density between metal grains. When the temperature is low, the dislocations are difficult to react and recombine with each other. Therefore, dynamic recrystallization is not easy to occur. But when the temperature rises, the heat activation effect of the atoms increase and the dislocation climb and cross-slip are easier to happen. It is conducive to dynamic recrystallization nucleation.

In addition, Fig. 3 also lists the comparisons between the different passes. Except the temperature, the amount of deformation also has a great effect on microstructure evolution. Figure 3 shows that, with the increase of extrusion passes, the deformation of the grains, LPSO phase and twins become more severely. The extrusion path taken by the equal channel extrusion is Bc, that is, the specimens were rotated 90° along the same direction between two consecutive passes. Thus, the direction of stress, LPSO phase flow direction, are perpendicular to the direction of the previous pass. Meanwhile, the criss-crossed twin are formed. Due to changes in shear stress direction, the alloy sample is subjected to shear stress in all directions, and LPSO phases begin to kink together.

Figure 4 is a schematic diagram showing the change of LPSO phase in DS-Mg98.5Zn0.5Y1 alloy after the equal channel extrusion. The LPSO phase in the grains experienced the following refining process: original fine lamellae → bent lamellae → zigzag but cracked lamellae → rectangular microcells. When the lamellae are bent to a large angel, the crack occurs along the transverse shear plane. This result is consistent with other related results.

Figure 5 shows the variations in texture under different passes at the temperature of 400°C. After 1-pass ECAP, the base texture was formed in the alloy, and the strength of the texture was 9.6. After the 2-pass extrusion, it can be seen that the base surface of the grain deflected. The direction changed from the shear stress direction to the extrusion direction. And the strength of the base texture increased from 9.6 to 12.3. After three passes of extrusion, the sample was rotated again, and the base surface deflected to the shear direction. But the strength of the texture were barely changed. The change of texture direction are in accordance with the path of ECAP.

Figure 6(a) shows the TEM photograph of the alloy in the vicinity of the long-period phase after three passes of the ECAP. It is clear that the LPSO is broken. A large number of dislocations gather around the LPSO phase. Figure 6(b) shows the EDS analysis of the alloy, it indicates that area A are mainly composed of three elements: Mg, Zn and Y, that is, the LPSO phase.
As can be seen from Fig. 7, due to the hindrance of dislocations by LPSO, dislocations are concentrated around the LPSO phase. This phenomenon has a great effect on dynamic recrystallization. For better understand the effect of LPSO on dynamic recrystallization evolution during hot extrusion, the schematic illustrations are presented in Fig. 7. As described previously, there are a large number of lamellar LPSO phase in DS-Mg$_{98.5}$Zn$_{0.5}$Y$_1$ alloy. During the deformation process, when the dislocation passed through a layer of LPSO phase, it will be plugged in front of the other LPSO phase. This will lead to the accumulation of dislocations. These aggregated dislocations will form dislocation cells. Afterwards, these dislocations rearranged and cancelled each other. The cell wall become sharper and form a dislocation network. As the progress of deformation, more dislocations enter the subgrain boundary. The orientation difference between the subcrystals gradually increase. In the end, the large angle grain boundaries are formed. Compared with the Mg–Zn–Y alloy with a small volume fraction of LPSO phase, the equal channel extrusion has a stronger effect on grain refinement in the DS-Mg$_{98.5}$Zn$_{0.5}$Y$_1$ alloy.

Figure 8 shows the yield strength (YS), ultimate compression strength (UCS), and elongation to failure of the alloy. It is apparent that the as-cast alloy possess the lowest YS (62 MPa), UCS (164 MPa), and ductility (elongation of 12%). After 1p-ECAP, the mechanical properties of alloy are greatly improved, but the elongation is reduced. After 3p-ECAP, the DS-Mg$_{98.5}$Zn$_{0.5}$Y$_1$ alloy shows a more than fourfold increase in YS (242 MPa) and

![Fig. 3 Microstructure of Mg$_{98.5}$Zn$_{0.5}$Y$_1$ alloy processed by ECAP.](image-url)
UCS (282 MPa). But the elongation decrease. The higher strengths of the ECAP samples are ascribed to the smaller grain size and kinked LPSO in alloy. In general, grain refinement is beneficial to the improvement of plasticity. However, it is interesting to point out that the plasticity gradually decreased with increasing ECAP passes. The main reason may be ascribed to the texture.

As described previously in Fig. 5, after one extrusion, the base texture was formed in the alloy. Base texture is parallel to the extrusion direction, that is, the c-axis and the extrusion direction are parallel. When the stress is parallel to the basal plane, the Schmid factor of the base surface is zero. The base surface is in a hard orientation, and the slip system can not be activated. Therefore, plasticity decreases.
4. Conclusion

The microstructure of as-DS-grown Mg_{98.5}Zn_{0.5}Y_{1} alloy were investigated by ECAP, and the dynamic recrystallization mechanism were analyzed. The results can be summarized as follows:

1. After directional solidified, the grain of Mg_{98.5}Zn_{0.5}Y_{1} were 600 µm on average and had uniform growth direction. There was a large number of lamellar structures in the grains.

2. Kink deformation was generated from the early deformation stage. Kink deformation is an important mechanism for homogeneous strain generation in these alloys. During multi-pass deformation, the lamellar 14H phase experienced a three-step refining process, kinking, zigzagging, and breaking. These broken LPSO phases hindered the movement of dislocations and facilitated the formation of dynamic recrystallization.

3. After 3-passes ECAP, the yield strength, ultimate compression strength of DS Mg_{98.5}Zn_{0.5}Y_{1} increased, but the ductility reduced. We conclude this phenomenon to the weakness of the base texture.

Fig. 6 (a) TEM images in Mg_{98.5}Zn_{0.5}Y_{1} alloy after 3-passes ECAP (b) EDS analysis of the alloy.

Fig. 7 The diagram of 14H-LPSO on recrystallization in the Mg_{98.5}Zn_{0.5}Y_{1} alloy during ECAP.
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