Evolution of Microstructure and Properties of ZYK530 Magnesium Alloy during Extrusion-Forging compound Forming

Fenghong Cao\(^*\), Chang Chen\(^1\) and Yaohui Xu\(^1\)

\(^1\) School of Physics and Electronic Engineering, Laboratory for Functional Materials, Leshan Normal University, Leshan, Sichuan 614004, China

\(^*\)Corresponding author’s e-mail: lscaofh2004@sina.com

Abstract. The evolution of microstructure and mechanical properties of ZYK530 Magnesium alloy during extrusion and die forging compound forming process were studied and analyzed. The results show that the as-cast ZYK530 alloy mainly consists of a-\(\text{Mg}\) matrix and I-\(\text{Mg}_3\text{Zn}_6\text{Y}\) and a small amount of W-\(\text{Mg}_3\text{Y}_2\text{Zn}_3\) and \(\text{Mg}_2\text{Zn}_3\) phases. During extrusion to die forging forming, the grain size of as-cast ZYK530 Magnesium alloy is refined and uniform under the action of three-dimensional compressive stress and high temperature deformation. The I-\(\text{Mg}_3\text{Zn}_6\text{Y}\) phase of ZYK530 magnesium alloy decrease relatively, while the distribution of W-\(\text{Mg}_3\text{Y}_2\text{Zn}_3\) is more obvious, a small amount of Z-\(\text{Mg}_{12}\text{Zn}_6\text{Y}\) and \(\text{Mg}_2\text{Zn}_3\) phases and undetermined phases can be observed in the matrix. Tensile test results show that the tensile strength, yield strength and elongation of extruded preformed specimens are increase by 32.6\%, 18\% and 18\% respectively compared with as-cast specimens. The mechanical properties of final forged specimens are improved by 9\%, 38.6\% and 23\% respectively compared with those of extruded preformed specimens. The fracture mechanism changes form as-cast cleavage fracture to mixed fracture mechanism of toughness and brittleness.

1. Introduction

As the lightest and environmentally friendly metal structure material, magnesium alloy has received more and more attention in energy saving and emission reduction and product weight reduction, and it is becoming more and more widely used in automobiles, ships, military industry, 3c and other fields\(^[1\sim4]\). Y, Zn and Zr are important alloying elements for improving the mechanical properties of magnesium alloys\(^[5\sim9]\). According to the related literature\(^[10\sim13]\), the Zn/Y mass ratio affects the number and distribution of the I-\(\text{Mg}_3\text{Zn}_6\text{Y}\) phase (the icosahedral structure), W-\(\text{Mg}_3\text{Y}_2\text{Zn}_3\) phase (cubic structure) and Z-\(\text{Mg}_{12}\text{Zn}_6\text{Y}\) phase (18R modulation structure) in the magnesium alloy matrix, wherein the plastic properties of the three phases are I-\(\text{Mg}_3\text{Zn}_6\text{Y}\) phase\(>W-\text{Mg}_3\text{Y}_2\text{Zn}_3\) phase\(>Z-\text{Mg}_{12}\text{Zn}_6\text{Y}\) phase, therefore, the selection of a suitable Zn/Y is an important way to control the mechanical properties of the alloy.

Deformation is an important way to change the properties of magnesium alloys\(^[13]\), by refining alloy grain shape deformation, changing the grain orientation, eliminating the internal stress of formation in the process of casting and extrusion forming. However, at present, there is not much research on the microstructure and properties of semi-continuous casting ZYK530 alloy during the extrusion forming process. It is reported\(^[8]\) that the mechanical properties at room temperature are excellent when the mass ratio of Zn/Y is 1.33. Therefore, in this paper, based on the research of alternative materials for automobile steering arm, the as-cast ZYK530 Mg alloy is taken as the...
research object, and the evolution of microstructure and mechanical properties of ZYK530 Magnesium alloy during extrusion and forging compound forming is discussed, which provides a certain experimental theoretical basis for further improvement in strength and ductility of Mg-Zn-Zr series alloy, and also provides a certain experimental basis for the research on the lightweight of Magnesium alloy in automotive materials.

2. Experimental methods and materials

The experimental alloys were prepared by semi-continuous casting method, and the raw materials were ZK60 magnesium alloy, pure Mg ingots, pure Zn ingots, Mg-Zr (containing Zr30%) and Mg-Y (containing Y30%) intermediate alloys. The experimental alloys were smelted in a crucible, and the ZK60 magnesium alloy was used as a base material. 5wt%Zn, 3wt%Y and 0.6wt%Zr were respectively added to Zn, Mg-Y and Mg-Zr intermediate alloy when the melt temperature reached 740℃, the melt was refined by Ar gas and then continuously cast into \( \Phi 112\text{mm}\times125\text{mm} \) ingots at 730℃. The composition of the alloys was analyzed by XRF(X-Ray Fluorescence) as shown in Table1.

The sample of extrusion preformed billet was prepared on XJ-800 T extruder after the ingot was solution treated at 400℃×12h, as shown in Fig.1. The extrusion temperature is 420℃, the extrusion ratio and speed are 16:1 and 17mm/s respectively, and then the extrusion pre-formed sample is put into the die cavity for final forging. The forging direction is consistent with the extrusion direction. The forging temperature is 440℃, the degree of deformation is 50%, and the forged speed is 5.6mm/s, as shown in Fig.1. The ASTM(B557M-06) gauge size is 25mm×6mm×3.5mm.

![Fig.1 Tensile Samples](image)

### Table 1 Chemistry composition of alloy (mass fraction: %)

| Alloy   | Zn   | Zr   | Y    | Mn   | Fe   | Si   | Ni   | Cu   | Mg   |
|--------|------|------|------|------|------|------|------|------|------|
| ZYK530 | 4.89 | 0.51 | 2.89 | 0.006 | 0.002 | 0.009 | 0.0026 |     | Bal  |

After pre-grinding and polishing the cast, extruded and forged samples, the metallographic samples were corroded by picric acid to prepare metallographic samples. The corroded time was for 4–10s. The metallographic structure of the samples was observed and analyzed by OM(Optical Microscope), XRD(X-ray Diffraction), SEM(Scanning Electron Microscope), EDS(Energy Dispersive Spectrometer). The mechanical properties of the samples were tested by universal tensile testing machine, and the fracture morphology of the samples was scanned by SEM, and the fracture mechanism was analyzed.

3. Experimental results and discussion

3.1. Microstructure Evolution of ZYK530 Magnesium Alloy

It can be seen from the Fig.2(a) that the grain size is uniform, the grain boundary bends like plum blossom, the average grain size is about 27μm, the black second phase particles are dispersed in the crystal; at the same time, there are lots of lamellar and fish-bone eutectic microstructures at the triangular grain boundary as show in Fig.3(a), a, b and c, and a large number of fine line microstructures are pointed from the gain boundary to the grain interior.

Fig.2(b) shows the microstructure of as-extruded ZYK530 Magnesium alloy. It can be seen from the picture that the large dendrites in the as-cast state have disappeared completely, but the large
grains distributed along the extrusion flow line can be seen, as shown in Fig. 2(b). The point “A” indicates that incomplete dynamic recrystallization is incomplete during the cast-extrusion preforming, which may be due to uneven deformation during the extrusion preforming. The flow rate of the metal near the center of the mold is faster than that of the metal near the mold, and supercooling degree of alloy liquid near die wall is high. Therefore, during the extrusion preforming, the metals in the center of the mold are too fast to form dynamic recrystallized grains, while dynamic recrystallization of the metal near inner wall of the mold is relatively complete, forming a distinct equiaxed crystal. According to the related literature[10], the deformation of magnesium alloy above 350 ℃ is high temperature deformation, and temperature is an important factor affecting the deformation of the alloy. During the high temperature deformation process, the nucleation rate of dynamic recrystallization of the alloy is controlled by thermal activation. The temperature has a great effect on improving the plasticity and deformation speed of the alloy, so the dynamic recrystallization of the alloy is obvious and the grain of the alloy is remarkably refined, and the average grain size is 2.87μm. Compared with the extruded state, the incomplete dynamic recrystallized grains in the alloy along the extrusion direction disappear completely after die forging, as shown in Fig.2(c), and the microstructure presented obvious distribution of drum streamline along the die forging direction.

In the process of die forging forming, the length direction of extruded billet remains unchanged, so the metal flow direction is perpendicular to the die forging direction, and the alloy grains turn along the direction of die forging deformation occurred at the same time and the average gain size is 2.87μm which indicates the improvement of the grain size of the alloy is limited under high temperature and large deformation conditions.

From the analysis of SEM and EDS in Fig.3(a)–(b), it can be seen that the mass ratio of Zn/Y is about 3.8. Combined with the XRD diagram of as-cast ZYK530 alloy in Fig.3(c), the alloy is mainly composed of 1-Mg3Zn6Y phase, α-Mg matrix, a small amount of W-Mg3Y2Zn3 phase and Mg2Zn3 phase.
Fig. 3 SEM + EDS + XRD of the A point
(a) A point SEM; (b) A point EDS; (c) XRD of as-cast ZYK530

Fig. 4(a) is the SEM morphology and EDS of as-extruded ZYK530 alloy. It can be seen from the diagram that a large number of precipitated second phases are precipitated along the extrusion direction in the alloy. The mass ratio of Zn/Y is 1.22. Combining with the XRD pattern of Fig. 5(a), it can be seen that the main alloying phases are mainly W-Mg3Zn1Y2 and Z-Mg12ZnY. No peak phases of I-Mg3Zn6Y and Mg2Zn3 phases have been observed. Fig. 4(c) is an enlarged view of the microstructure of the die-forged ZYK530 Magnesium alloy of Fig. 4(b). During high temperature die forging, the number of second phases precipitated along the direction of forging deformation increases obviously under the action of three-dimensional compressive stress. According to EDS in Fig. 4(c), the mass ratio of Zn/Y is about 1.43. In addition to I-Mg3Zn6Y, W-Mg3Y2Zn3, there are a small number of Mg3Zn3 and Z-Mg12ZnY phases and uncertain phases in the XRD, as shown in Fig. 5(b).

Fig. 5 XRD of the as-extruded and as-forged ZYK530 magnesium alloy
3.2. Discussion and analysis

According to the literature, when the mass ratio of Zn/Y is greater than 4.38, the quasicrystalline phase I-Mg$_3$Zn$_6$Y is easily formed by conventional casting; when the mass ratio of Zn/Y is less than 1.10, the main phase is W-Mg$_3$Zn$_3$Y; while when the mass ratio of Zn/Y is 1.10–4.38, the main phases in the alloy are I phase and W-Mg$_3$Zn$_3$Y$_2$ phase[14]. From the experimental results, it can be seen that the phase composition of as-cast to as-extruded forming is inconsistent with that reported.

In the process of semi-continuous casting, according to the phase diagrams of Mg-Zn and Mg-Y binary alloy[15], the solid solubility of Y in Mg is higher than that of Zn, and it is easy to form segregation at the grain boundary to form compound phase. That is to say, during the semi-continuous casting process, α-Mg precipitates first, then α-Mg+W-Mg$_3$Zn$_3$Y$_2$ is formed under eutectic condition. With the continuation of solidification, the alloy undergoes peritectic reaction and forms α-Mg+W-Mg$_3$Zn$_6$Y, because of uneven crystallization in the casting process. In the presence of equilibrium segregation, there may be a small amount of residual peritectic reaction, namely W-Mg$_3$Y$_2$Zn$_3$ phase and Mg$_2$Zn$_3$ phase. The I-Mg$_3$Zn$_6$Y phase has good coherence with Mg matrix, but I-Mg$_3$Zn$_6$Y phase has a larger volume and a certain distribution of W-Mg$_3$Y$_2$Zn$_3$ phase, so the mechanical properties of as-cast ZYK530 alloy is poor.

The I-Mg$_3$Zn$_6$Y and Mg$_2$Zn$_3$ peaks were not observed in the extruded XRD pattern. The reason may be that the large block I-Mg$_3$Zn$_6$Y is crushed into small particles under the action of the triaxial compressive stress, and forms the core of recrystallization, the I-Mg$_3$Zn$_6$Y phase is completely dissolved in the matrix, and the Y supersaturation diffused into the matrix tends to form a supersaturated solid solution, so that excess Y precipitates, forming a W-Mg$_3$Zn$_3$Y$_2$ phase with Mg and Zn according to an atomic ratio, and the Z-Mg$_{12}$Zn$_Y$ phase is easily formed when the mass ratio of Zn/Y is very low, therefore, it consists of W-Mg$_3$Zn$_3$Y$_2$ phase in as-extruded alloy and distributes a small amount of Z-Mg$_{12}$Zn$_Y$ phase. In the process of die forging, the phase of the alloy is decomposed during the high temperature and three-direction stress deformation, and I-Mg$_3$Zn$_6$Y phase, W-Mg$_3$Zn$_3$Y phase and Z-Mg$_{12}$Zn$_Y$ phase are decomposed, while the I-Mg$_3$Zn$_6$ phase reinforcing phase is retained, indicating that the alloy is in the process of high temperature deformation and cooling. The Zn and Y atoms are redistributed, and the crushed grains are also used as the core of the recrystallized nucleus while forming a small amount of W-Mg$_3$Zn$_3$Y$_2$ phase, Z-Mg$_{12}$Zn$_Y$ phase, Mg$_2$Zn$_3$ phase and a small amount of undetermined phase.

In summary, dynamic recrystallization occurs during deformation when the deformation temperature of the alloy exceeds 1/3 of the melting point of the alloy[16]. During the process from casting to forging, besides obvious dynamic recrystallization, there are also obvious rearrangements of Zn and Y atoms, which makes the volume fraction and distribution of I-Mg$_3$Zn$_6$Y, W-Mg$_3$Zn$_3$Y and Z-Mg$_{12}$Zn$_Y$ change obviously. The mechanical properties of the alloy will be affected by melting, and the grain size does not change significantly from extrusion to forging, which indicates that the effect of high temperature and large deformation on grain size is limited.

3.3. Mechanical properties analysis

Fig.6 shows the tensile properties under different forming conditions. The results show that the tensile strength $\sigma_b$ and yield strength $\sigma_s$ of ZYK530 magnesium alloy are increased by 44.5%, 63.5% and elongation $\delta$ by 8.8%, respectively.
Fig. 6 Mechanical properties of ZYK530 Alloy under Different Conditions

With the change of deformation mode, the yield strength of the alloy increases significantly, mainly due to the change of grain size and the change of deformation strengthening mechanism. ZYK530 magnesium alloy undergoes obvious continuous dynamic recrystallization during extrusion preforming and die forging, and the grain size is difficult to grow. At the same time, the second phase particles also hinder the dynamic crystal growth of the alloy. Therefore, compared with as-cast ZYK530, the phase of the extrusion preformed alloy is mainly composed of W-Mg3Zn3Y2 and Z-Mg12ZnY, in which W-Mg3Zn3Y2 has weaker bonding ability with magnesium matrix and deteriorates the mechanical properties of the alloy; Z-Mg12ZnY phase is an unstable phase, but it helps to improve the properties of the alloy, and its mechanical properties are better than those of as-cast alloy. The results show that grain refinement plays a decisive role in the mechanical properties of the alloy. In addition to W-Mg3Zn3Y2 phase and Z-Mg12ZnY phase, I-Mg3Zn6Y phase and a small amount of Mg2Zn3 phase in the alloy which have strong bonding force with magnesium matrix are added to the alloy during the final forging process, which in conducive to further improving the properties of alloy. Although the grain size of the die-forged specimen does not change significantly compared with that of the extruded specimen, the mechanical properties of the die-forged specimen are also improved significantly compared with that of the extruded specimen. The Mg-Zn-Y phase transformation of ZYK530 alloy is complex in the process of casting-extrusion-forging compound forming, and the I-Mg12ZnY phase is not easy to move, which can effectively hinder the movement of dislocations and improve the mechanical properties of the alloy. Grain refinement is the decisive factor for the improvement of mechanical properties of ZYK530 alloy. The mechanical properties of ZYK530 magnesium alloy are obviously improved after extrusion-forging compound forming.

3.4. Fracture analysis

Fig. 7 shows the tensile fracture morphology of the ZYK530 magnesium alloy in different states. The ZYK530 magnesium alloy has a jagged fracture with no obvious necking, and the tearing edge is the main way to connect the cleavage facets and form the cleavage river. At the same time, white precipitates were observed on the grain boundaries, indicating that the fracture mechanism was mainly brittle fracture, as shown in Fig. 7(a). After extrusion-forging compound forming, the macro-fractures have obvious necking, and there are obvious dimples on the fractures. The dimples on the die forged sample fracture surface increase obviously and are uniform and small, and distribute in the streamline along the forging direction, as shown in Fig. 7(b)–(c), which shows that the dimples on the extrusion fracture surface distribute along the extrusion direction. In addition, fine white particles precipitate on the fracture surface of as-extruded and die forged specimens, and there are a few cleavage steps, so the fracture mechanism is ductile-brittle mixed fracture. The results show that the effect of deformation on
grain size is limited during extrusion forging process, but it has a great influence on the volume fraction and phase distribution of the alloy, which affects the mechanical properties of the alloy.

Fig. 7 Fracture morphology of ZYK530 magnesium alloys in different states

4. Conclusion

1) The I-Mg₃Zn₆Y and W-Mg₃Y₂Zn₃ phases and a small amount of Mg₂Zn₃ phase in the as-cast ZYK530 alloy are mainly distributed at triangular grain boundaries and the grain size is relatively coarse. After the extrusion-forging compound forming, the dynamic recrystallization of ZYK530 alloy under three-dimensional stress makes the grain refined obviously, and the number and distribution of precipitated I-Mg₃Zn₆Y, Z-Mg₁₂Zn₁₆Y phase, W-Mg₃Y₂Zn₃ phase and Mg₂Zn₃ phase undergo complex changes, which play an important role in the mechanical properties of the alloy, but the mechanical properties a key to the role is grain refinement.

2) The as-cast ZYK530 magnesium alloy is obviously a brittle fracture mechanism; there are more and more fine dimple fracture characteristics as-forged state than as-extruded state. In the former, in the former, equiaxed dimples are mainly distributed with small quasi-cleavage surface and tearing edges, which belong to the mixed fracture characteristics of toughness and brittleness, while in the latter, equiaxed dimples are mainly distributed. In the latter, the fracture is an equiaxed dimple morphology, which belongs to the ductile fracture mechanism.

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