Effects of Y Content on the Microstructures and Mechanical Properties of Mg-5Zn-xY-0.6Zr Alloys

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Abstract: Three as-cast and as-extruded Mg-5Zn-xY-0.6Zr (x=5 wt%, 8 wt%, 11 wt%) alloys were prepared, and the effects of Y content on the microstructures and mechanical properties of the alloys were investigated. The results show that the investigated Mg-Zn-Y-Zr alloys mainly consist of α-Mg, X-Mg_{12}YZn and minor amount of W-$Mg_3Y_2Zn_3$ phases. The volume fraction of X-Mg_{12}YZn phase increases and that of W-$Mg_3Y_2Zn_3$ phase decreases with the rising of Y content in the alloys. The as-extruded Mg-5Zn-11Y-0.6Zr alloy owns the optimal ultimate tensile strength and yield strength of 429 and 351 MPa, respectively. Mg-5Zn-5Y-0.6Zr alloy owns the maximum elongation of 13.6%.

Key words: Mg-Zn-Y-Zr alloy; indirect extrusion; ternary phase; mechanical property

1 Introduction

Magnesium alloys are one of the lightest structural metallic materials which got the effective applications in the industries[1,2]. They own low density, high specific strength, specific stiffness and good damping capacity. Recently, they have been increasingly attracting scientific attentions due to the demand for the weight reduction of auto-mobiles and air crafts to lower the CO_2 emission and to enhance the fuel efficiency[3-6]. It has been known as the "the most important environmental structural metallic materials in the 21 century"[7].

Magnesium alloys are divided into Mg-Al, Mg-Mn, Mg-Zn and Mg-RE, *etc*, according to elemental addition to magnesium alloys at present. Magnesium alloys with rare earth elements(RE) addition are known for the excellent performance at the elevated temperature as well as the room temperature, which owing to the formation of high strength and heat-resistant compounds[8-10]. However, large amount of RE addition causes plenty of excess cost. What is more, the production process will be more complex due to the addition of a large number of rare earth elements. So to develop a new and low cost of heat resistant magnesium alloy has become an important issue in magnesium alloy fields. The Mg-Zn-Y heat resistant magnesium alloys have been developed in this context[11,12]. Y is one of the elements that owns the largest equilibrium solid solubility in Mg matrix, up to 12.5 wt%. It has been reported that Y addition in magnesium alloys could refine the grains and improve the aging response of Mg-RE alloys. Furthermore, Y element is beneficial for the as-cast Mg alloys because of its outstandingsolid solution strengthening effects and good flame retardant effects[13-17]. Many researches have shown that[18-22] three ternary equilibrium phases exist in Mg-Zn-Y system, one is W-phase ($Mg_3Y_2Zn_3$), with cubic structure; another is I-phase (Mg_3Zn_6Y), which is icosahedral quasicrystal structure first reported by Luo Z P *et al*; the third one is X-phase (Mg_{12}ZnY), long period stacking ordered structure. It revealed that the increasing volume fraction of X-phase could improve the mechanical properties of the as-extruded Mg-Zn-Y alloys. Meanwhile too much icosahedral quasicrystal structure I-phase and network eutectic W-phase brought disadvantageous influence on mechanical properties of Mg-Zn-Y alloys[23]. It still

Journal of Wuhan University of Technology-Mater. Sci. Ed. www.jwutms.net Feb. 2019

139

requires more efforts to strengthen the magnesium alloys by taking advantages of the prospecting X-phase and avoiding redundant I-phase and W-phase.

We designed three types of Mg-Zn-Y-Zr alloys with different Y contents, in order to investigate the effects of phase composition and content on the microstructures and mechanical properties of Mg-Zn-Y-Zr alloys. Besides, the Zr addition is aimed at grain refinement for the designed alloys.

2 Experimental

Mg-Zn-Y-Zr alloys were prepared from high-purity (99.9%) Mg, pure Zn (99.9%), Mg-50Y and Mg-30Zr master alloys by induction melting in a mild steel crucible at 710 ℃ and casting into a preheated steel mold (about 200 ℃) under mixed SF_6 and CO_2. Flux was used to purify the melt, as well as prevent from burning. The casting ingots were 60 mm in diameter and 120 mm in height. After casting process, the ingots were homogenized at 510 ℃ for 16 h. Then the ingots were machined into a diameter of 47 mm cylinders with a length of 100 mm, and were indirectly extruded into 12 mm rods with a speed of 56 mm/s in a diameter of 50 mm extruding container, at 400 ℃. Meanwhile, the extrusion ratio was 15:1, and the container and mold were preheated to 350 ℃. The rods were machined into tensile specimens of 6 mm gauge diameter and 25 mm gauge length, according to the ASTM standard B557M-10. The tensile direction was parallel to the extrusion direction. Tensile test was performed at room temperature using a Shimadzu AG-X (10kN) machine (the speed of tensile was 0.017 mm/s).

Table 1 Actual chemical compositions of the investigated alloys

Alloys	Nominal composition	Actual composition/wt%			
		Zn	Y	Zr	Mg
WZ505	Mg-5Zn-5Y-0.6Zr	4.77	5.68	0.566	Bal.
WZ805	Mg-5Zn-8Y-0.6Zr	5.10	8.99	0.498	Bal.
WZ1105	Mg-5Zn-11Y-0.6Zr	5.70	13.7	0.378	Bal.

The as-cast and as-extruded spicemen were mechanically polished and etched with a 4% nital and an ethanol solution of picric acid and glacial acetic acid (2 g picric acid, 5 mL glacial acetic acid, 5 mL water and 25 mL ethanol), respectively. Microstructures of the alloys were observed by optical microscope (OM), scanning electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDS). The phase analyses were performed with an X-ray diffractometer (XRD), and the scanning angle was from 15 to 75° with a speed of 0.13°/s. The XRD patterns were indexed using PDF standard card (2004). Actual compositions of the prepared alloys were obtained by chemical analysis (an inductively coupled plasma mass spectrometry) as listed in Table 1.

3 Results

3.1 Microstructures of the as-cast alloys

Fig.1 shows the optical microstructures of as-cast and as-homogenized Mg-5Zn-xY-0.6Zr (x=5 wt%, 8 wt%, 11 wt%) alloys. It can be seen that grey and white block phases in three alloys. Whereas, the variation of Y significantly affects the distribution and volume fraction of each phase. The secondary phase forms a three-dimensional discontious honeycomb-like network
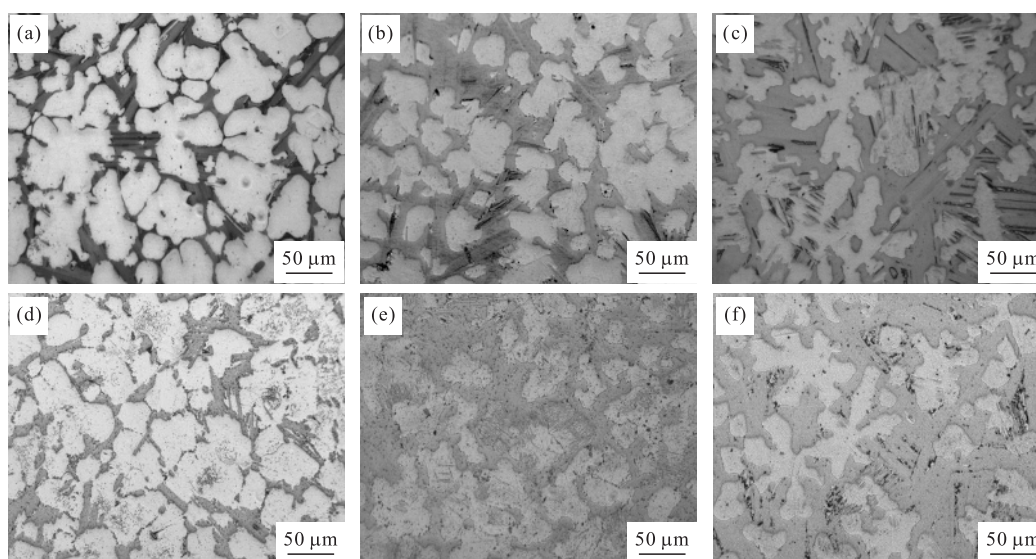

Fig.1 Microstructures of the as-cast and as-homogenized Mg-5Zn-xY-0.6Zr alloys: (a), (d) as-cast and as-homogenized WZ505; (b), (e) as-cast and as-homogenized WZ805; (c), (f) as-cast and as-homogenized WZ1105

at Magnesium grain boundaries of as-cast Mg-5Zn-5Y-0.6Zr alloy as shown in Fig.1(a). With increasing the addition of Y to 8.99%, reticular eutectics honeycomb-like secondary phase gradually growing into tabular and flaked ones, while the phase species unchanged as shown in Fig.1(b). In addition, with Y content increased from 8.99 wt% to 13.7 wt%, the second phases transformed into thick and compact plate-like patterns as clearly shown in Fig.1(c). The secondary phases almost completely taken up the considerable area in α-Mg matrix. It illustrates that the microstructures of as-cast alloys all exhibit typical dendritic structure as shown in Fig.1. After homogenization treated at 510 ℃ for 16 h, most of the eutectic compounds at the grain boundaries partially dissolve into α-Mg matrix, and the remnant particles are discontinuously distributing throughout the matrix as shown in Fig.1(d), (e) and (f), respectively.
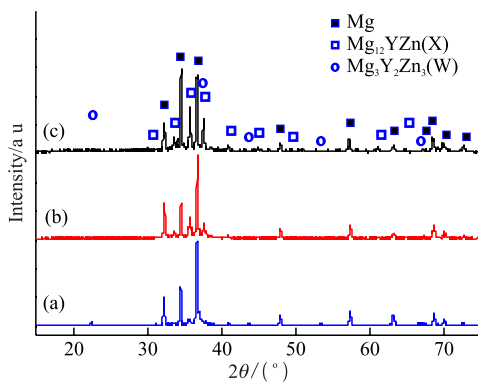

Fig.2   XRD patterns of as-cast Mg-Zn-Y-Zr alloys: (a) WZ505; (b) WZ805; (c)WZ1105

Fig.2 shows the XRD patterns of as-cast alloys. It suggests that WZ505 alloy consists of α-Mg solid solution, $X\text{-}Mg_{12}YZn$ and $W\text{-}Mg_3Y_2Zn_3$ phases. As Y content increased to 8.99 wt%, $W\text{-}Mg_3Y_2Zn_3$ phase disappeared, $X\text{-}Mg_{12}YZn$ phase became the main secondary phase in WZ805 alloy. Simultaneously, the diffraction peaks intensity of $X\text{-}Mg_{12}YZn$ phase is stronger with the increased Y. This result is not in conformity with the researches done on Mg-Y-Zn-Zr system alloys[24], that is due to little volume fraction of $W\text{-}Mg_3Y_2Zn_3$ phase is hardly identified out in XRD patterns. With the Y addition further increased to 13.7 wt%, $W\text{-}Mg_3Y_2Zn_3$ phase disappeared completely. Compared to each respective neighbor peaks for α-Mg, the X-phase intensity of the diffraction peaks in WZ1105 alloy was strongest. It suggests that the volume content of X-phase increased with the addition of Y, which is consistent with phase analysis.

In addition, Zr addition has no effect on the phase composition of Mg-Zn-Y-Zr alloys[25].

It requires a brief discussion to understand the distribution and volume fraction of X- and W-phase. In the conventional non-equilibrium solidifying process, if one assumes no diffusion in the solid but perfect mixing in the liquid, the mean composition of the solidified α-Mg crystals is always lower than the nominal composition of the alloy, and the element Y and Zn are pushed into the liquid next to the solid-liquid interface. With the nucleation and growth of primary α-Mg in progress, more and more solute Y and Zn are distributing in the liquid around the primary α-Mg crystal nucleus. Furthermore, due to the decreasing of temperature with the solidification further in progress, the solute diffusion rate drops. It is quite possible that the concentration fluctuation for Y and Zn solute could take place in the localized micro-areas. The reaction temperature for X-phase is 20 ℃ higher than that for W-phase in Mg-Y-Zn systems[26]. X-phase is prior to W-phase for precipitation in this sense. With the solidifying sequence, the amount of liquid decreases so that there are no sufficient Mg atoms to participate in the precipitation of X-phase. Hence, the appearance for W-phase is quite reasonable with the precipitated X-phase. With the increasing content of solute Y, it is more close to generate the $X\text{-}Mg_{12}YZn$ phase which Zn/Y atom ratio is 1. On the other hand, it needs more Zn to form $W\text{-}Mg_3Y_2Zn_3$ phase because Zn/Y atom ratio is 1.5. So it should be noted that the volume fraction of W-phase decreases but the amount of X-phase increases with the increasing Y additions.

Table 2 EDS results of as-cast alloys

Position	Mg/at%	Zn/at%	Y/at%	Y/Zn（Atom ratio）
A	96.745	0.984	2.271	—
B	53.109	27.475	19.416	0.71
C	86.966	5.523	7.511	1.36

Fig.3 shows the SEM images and EDS analysis of as-cast Mg-5Zn-xY-0.6Zr alloys. It illustrates that the reticular intermetallics is made of Mg, Zn and Y in position B. Also the atomic ratio of Y and Zn is nearly 2:3, which is corresponding to the XRD result of $W\text{-}Mg_3Y_2Zn_3$ phase shown in Fig.2. Therefore, the reticular intermetallics phase should be $W\text{-}Mg_3Y_2Zn_3$. Similarly, EDS result of the gray flaked phase in position C is also shown in Table 2. The atomic ratio between Y and Zn is approximately 1:1, which is in accordance with $X\text{-}Mg_{12}YZn$ phase shown in Fig.2.

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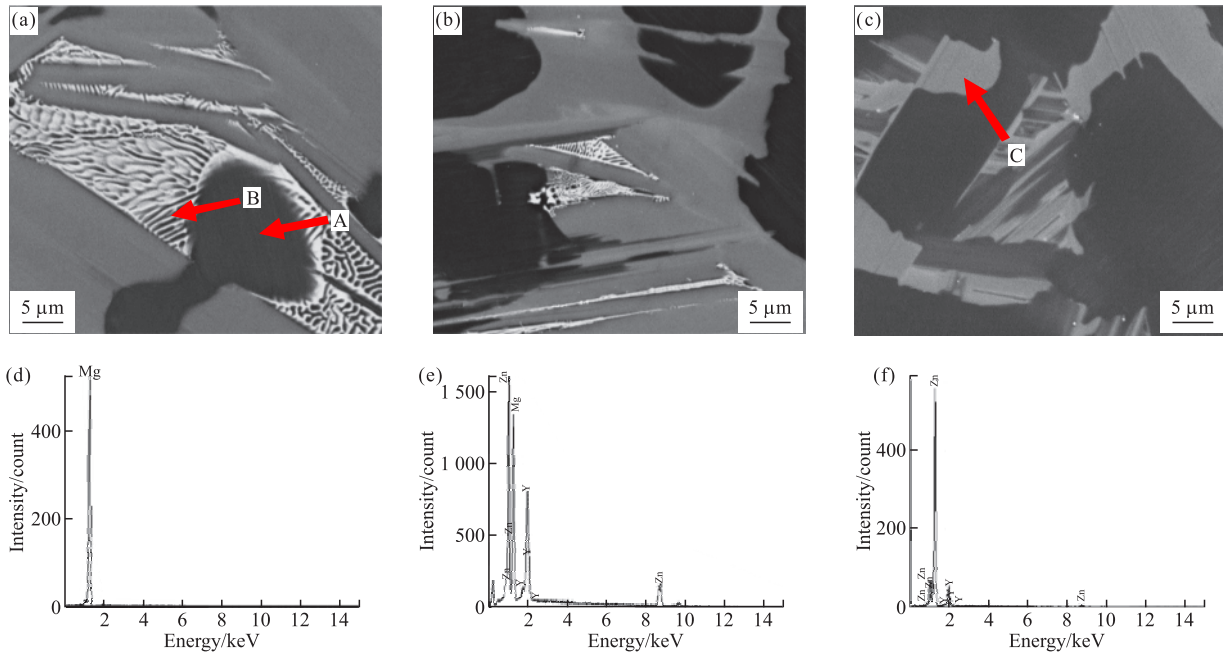
141

Fig.3 SEM images of (a) WZ505; (b) WZ805; (c) WZ1105; EDS of (d) Position A; (e) Position B; (f) Position C
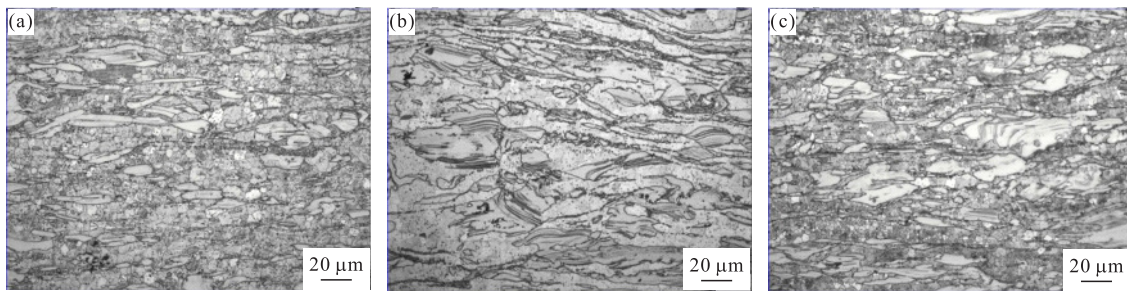

Fig.4 Microstructures of as-extruded Mg-Zn-Y-Zr alloys: (a)WZ505; (b) WZ805; (c) WZ1105

Thus, with consideration of the EDS analyses, it can be concluded that WZ505 alloy consists of α-Mg, W-$Mg_3Y_2Zn_3$ and X-$Mg_{12}YZn$ phases. WZ805 alloy consists of α-Mg, X-$Mg_{12}YZn$ phase and minor W-$Mg_3Y_2Zn_3$. Meanwhile, only α-Mg and X-$Mg_{12}YZn$ phase exist in WZ1105 alloy. These results are in good agreement with XRD patterns.

3.2 Microstructures of the as-extruded alloys

Fig.4 shows the optical microstructures of the as-extruded alloys on longitudinal section along extrusion direction. It could be generally seen that the secondary phases were all well-distributed. What is more, the lamellar X-phase was cracked, elongated and oriented along the extrusion direction. Meanwhile, no parallel flow lines appeared along the extrusion direction of the three alloys. The X-ray diffraction patterns of as-extruded Mg-5Zn-xY-0.6Zr alloys are shown in Fig.5. However, minor amount of equiaxed grains were observed behind the destroyed compounds, which

indicated that dynamic recrystallization (DRX) occured during hot extrusion. Thus, the microstructures of the alloys have been clearly refined by the following indirect extrusion. With the additive amount of Y increased, the volume fraction of second phase significantly increased and then changed little.

Compared to the as-cast microstructures, reticular eutectics rose-shape second phase distributed at the grain boundaries vanished as clearly shown in Fig.4(a). Meanwhile, the second phases performed more uniformly, and the inhomogeneity of dynamic recrystallization grain size was also displayed in Fig.4. It indicates that the cracked phase has significant effect on the nucleation and growth of DRX grains.

The X-ray diffraction patterns of as-extruded Mg-5Zn-xY-0.6Zr alloys are shown in Fig.5. SEM micrographs and corresponding EDS microanalysis of the alloys are presented in Fig.6, respectively. It suggests that the phase composition of WZ505 and WZ805 alloys are mainly α-Mg solid solution,
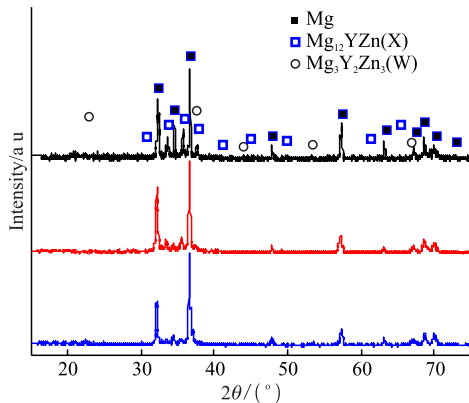
Fig. 5 XRD patterns of as-extruded Mg-Zn-Y-Zr alloys: (a) WZ505; (b) WZ805; (c) WZ1105

Table 3 EDS results of the as-extruded alloys

Position	Mg/at%	Zn/at%	Y/at%	Y/Zn (Atom ratio)
A	98.596	0.489	0.915	—
B	53.109	27.475	19.416	0.71
C	65.065	15.477	19.458	1.257

W-$Mg_3Y_2Zn_3$ and X-$Mg_{12}YZn$ phases. However, the distribution of W-phase is more obvious around the stripped phases than in matrix. Moreover, the diffraction peaks intensity of W-$Mg_3Y_2Zn_3$ phase is especially weak. The result illustrates that volume fraction of W- phases is little in WZ805 alloy. There are only α-Mg and X-$Mg_{12}YZn$ phase shown in Fig.5(c), it proves W-$Mg_3Y_2Zn_3$ phase doesn't exist in WZ1105 alloy. Results suggest that phase compositions have no obvious changes after indirect extrusion, but the phase morphology changes a lot in the process. It could be confirmed by the EDS analysis that position B in Fig.6(a) shown in Table 3 illustrates the atomic ratio

between Y and Zn is nearly 2:3 in both large and small particles. This is corresponding to the XRD results of W-$Mg_3Y_2Zn_3$ phase shown in Fig.4. The EDS result of position C indicates that the atomic ratio of Y and Zn is approximately 1:1, it reveales the stripped fiber-like phase refers to X-$Mg_{12}YZn$ in Fig.6(c).

3.3 Mechanical properties of the as-extruded alloys

Fig.7 shows the mechanical properties of the as-extruded Mg-5Zn-xY-0.6Zr alloys, including ultimate tensile strength, tensile yeild strength and elongation at room temperature. It is worthy mentioning that the elongation of the alloys is generally decreased with the rising Y content. However, the yield strength and tensile strength increase rapidly with the increasing Y addition.

For WZ505 alloy with low Y content whose mechanical property is the lowest of the three alloys, the elongation is 13.6%, reaching the maximum value. While its yield strength and tensile strength are 215 and 327 MPa, respectively. With the increasing addition of Y, the strength of alloys improves greatly. The yield strength and tensile strength of as-extruded WZ805 alloy are 256 and 381 MPa, respectively, but its elongation decreases to 7.7% as shown in Fig.7(b). It shows that yield strength and tensile strength can increase by 19% and 17%, respectively. Compared with WZ505 alloy, the mechanical properties of WZ1105 alloy increased greatly with the highest Y content. It can be seen that the yield strength and tensile strength are 351 and 429 MPa of WZ1105 alloy, increased by 37% and 13% separately compared with WZ505 alloy.
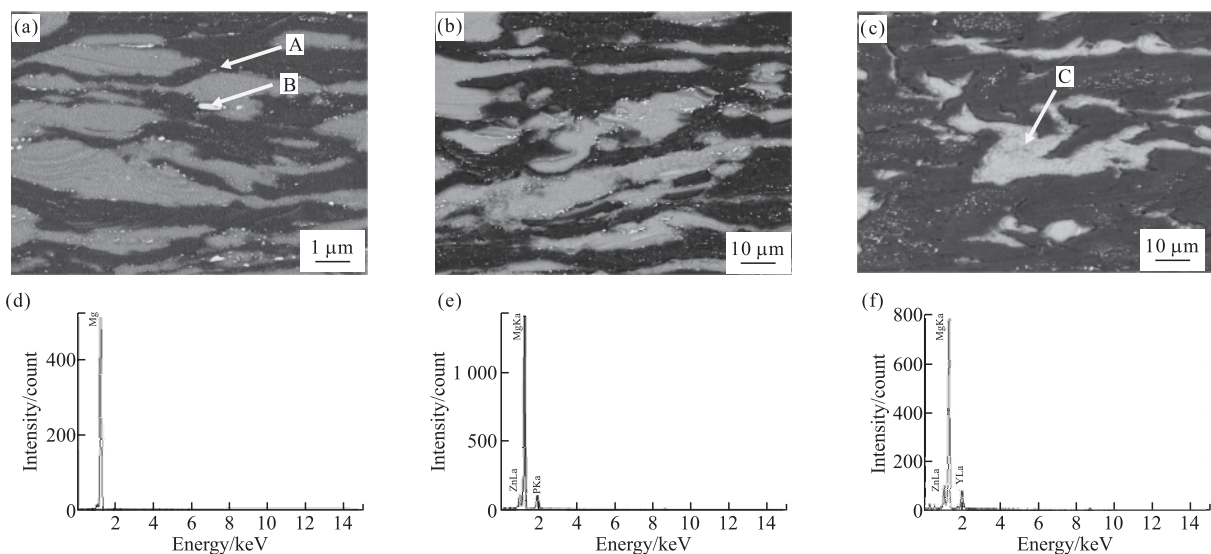

Fig.6 SEM images of as-extruded Mg-Zn-Y-Zr alloys: (a) WZ505; (b) WZ805; (c) WZ1105; EDS of (d) Position A; (e) Position B; (f) Position C
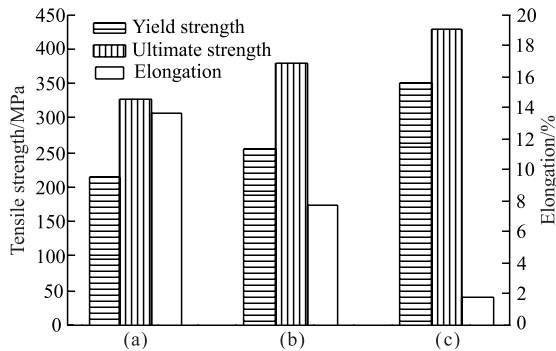
Journal of Wuhan University of Technology-Mater. Sci. Ed. www.jwutms.net Feb. 2019

143

Fig.7 The tensile properties of the as-extruded alloys: (a)WZ505; (b) WZ805; (c) WZ1105

Similarly, the elongation reaches the mimimum of the three experimental alloys, that is just 1.8%.

4 Discussion

With the concentration of Y content increased from 5.68 wt% to 13.7 wt% in Mg-5Zn-xY-0.6Zr alloys, the composition of phases changed from α-Mg solid solution, X-Mg_{12}YZn and W-$Mg_3Y_2Zn_3$ phases to α-Mg solid solution and X-Mg_{12}YZn phases, W-phase disappeared. Zn and Y elements are very important to Mg-Zn-Y-Zr alloys. They added into magnesium alloys and generally exist in two forms, one is to solve into the α-Mg matrix, so that the alloy produces solid solution strengthening effect; the other is to form the secondary phase that playes in second phase strengthening effect. The solid solubility of Zn and Y in magnesium alloys is separately 6.2 wt% and 12.5 wt%. In the solidifying process, Y and Zn elements solubilized in α-Mg matrix and well-distributed. Then the solute Y and Zn are pushed into the liquid next to the solid-liquid interface to form the second phases at the grain boundaries. It also manifests that despite the solid solubility of Y element in the alloys is high, there are inevitably second phases exist in non-equilibrium solidification process. The reason is that with the nucleation and growth of primary α-Mg in progress, more and more solute Y and Zn are distributing in the liquid around the primary α-Mg crystal nucleus. At the final solidifying stage, the concentration of Zn and Y elements is high enough to form the second phases at grain boundaries. Secondary phase strengthening is the primary strengthening mechanism of the alloys. The reason is that three experimental alloys present different Mg-Zn-Y ternary phases in different conditions. The compositions and components of the phases are related to Y/Zn mole ratio. The Y/Zn concentration ratio leads to the different compositions of the phase. The detailed discussion is described in Reference[14].

Tensile results show different Y contents have significant influence on the mechanical properties of as-extruded Mg-Zn-Y-Zr alloys considerably. For the alloys with increasing addition of Y, the volume fraction of X-phase increases rapidly. Result demonstrates that X-phase has an important reinforcement effect in magnesium alloys. Firstly, the increasing amount of X-phase could have a refining effect on the dynamic recrystallized grains. According to the Hall-Petch equation, yielding strength could be enhanced owning to the grain refinement. Secondly, X-phase is lamellar elongated and oriented along the extrusion direction, that could be regarded as strengthening fibers in composite materials. Meanwhile, the volume fraction of W-phase reduced until vanished with the increasing of Y. The phenomenon demonstrates W-phase is against for the strength in Mg-Zn-Y-Zr alloys. It requires a brief discussion to understand the principle why W-phase is harmful to the mechanical properties of the alloys. In the previous investigation by Singh that W-phase is net-like microstructure which is destructive to the mechanical properties. In addition, higher stress concentration will more easily occurr around the particles with larger size. When the volume fraction is high enough to cause the brittle fracture of net-like distributed W-phase, it is more sensitive to the stress concentration. The mechanical properties will be degraded by the net-like microstructure. Elongation decreases because of the suppression of the dislocation movement. Because the phase in WZ505 alloy is fine and uniformly dispersed in matrix, the displacement of dislocation glide is sharply reduced in this alloy during tensile process. So the plasticity of the WZ505 alloy is much higher.

5 Conclusions

Mg-5Zn-0.6Zr alloys with different Y contents from 5 wt% to 11 wt% were synthesized and characterized. Accordingly, the following conclusions can be drawn:

a) Three investigated Mg-Zn-Y-Zr alloys own an alternant distribution of X-phase (Mg_{12}YZn) lamella and W-phase(MgY_2Zn_3) eutectics. With the increasing Y addition, the volume fraction of X-phase increases, and the W-phase disappears. Simultaneously, the distribution transforms from slender areolar tissue to broad lamellar structure.

b) During extrusion, all the secondary phases are

well-distributed, and no parallel flow lines appeared along the extrusion direction. The reticular eutectics rose-shape second phases which distributed at the grain boundaries vanished, and the secondary phases unchanged in the extruded process.

c) Owing to the grain refinement and reinforcement by X-phase, the as-extruded WZ1105 alloy owns the maximum tensile and yielding strength of 429 and 351 MPa, respectively. As well as WZ505 alloy containing W-$Mg_3Y_2Zn_3$ phase owns the optimum elongation, which is 13.6%.

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