Effects of Sm addition on microstructural evolution of Mg-6Zn-0.4Zr alloy during semi-solid isothermal heat treatment

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Abstract: The application of segmental semi-solid thixoforming of magnesium alloys is confined due to the dimensional distinction existing in solid particles of the alloy billet from edge to center zones. In the present study, the effects of Sm addition on the microstructural evolution of Mg-6Zn-0.4Zr and Mg-6Zn-4Sm-0.4Zr alloys by semi-solid isothermal heat treatment were investigated, to obtain optimum semi-solid microstructures for the subsequently thixoforming. The results indicate that the grains of the Sm-bearing alloy are evidently refined and gradually evolve from dendritic to globular and elliptic particles. In addition, the distinctly dimensional effect of the Mg-6Zn-0.4Zr alloy is eliminated with 4% Sm addition; the particle sizes in all zones from center to the edge of the billet are almost identical. With the increment of isothermal heat treatment temperature, the dendritic microstructures completely disappear, and meanwhile, the irregular and globular particles gradually form. The size, morphology and the distribution of solid particles mainly depend on the formation and permeation of the liquid phase in the process of isothermal heat treatment. As the isothermal temperature increases from 570 °C to 610 °C, the average size and shape factor of solid particles of both the alloys with and without Sm addition gradually decrease while the liquid fraction gradually increases.

Key words: Mg-Zn-Zr magnesium alloy; dimensional effect; Sm; semi-solid isothermal heat treatment; non-dendritic microstructure

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Magnesium alloys are one of the most important structural materials with high specific strength and stiffness, low density, excellent castability, remarkable electromagnetic shielding effect, and fine machining-ability. For these reasons, magnesium alloys become increasingly attractive for applications in the automotive and aerospace industries^[1-2]. However, the application of magnesium alloy castings processed through traditional technologies is still very limited due to their hot cracking tendency, serious micro-porosities and low mechanical properties. In addition, magnesium alloy melt can be extremely flammable and explosive in the process of industrial production. What is even worse from micro-aspects would be that the Mg-Zn binary alloys have coarse dendritic microstructure, leading to high tendencies for hot cracking, critical shrinkage

cavities and shrinkage porosities. These disadvantages will affect the mechanical properties of the alloys^[3] so much that the mechanical properties cannot meet the requirements in many applications. Therefore, how to improve microstructure of magnesium alloys is a difficult task to the researchers. In order to extend the application of Mg-Zn binary alloys, it is necessary to add alloying elements to refine the grains, or using innovative processing methods. Since Flemings et al.^[4] found that non-dendritic structures have special properties in the 1970s, semi-solid forming technology has been rapidly developed as a new forming technology^[5]. There are two ways for semi-solid forming: the rheological forming and thixoforming^[6-7]. Compared with traditional casting and forging methods, thixoforming is a relatively new metal forming technology and has many advantages. As a key branch of semi-solid technology, thixoforming has attracted much attention due to its technical and economic advantages in processing alloys, which comprises of preparation, partial remelting and thixoforming of semi-solid billets. The preparation of semi-solid billet is the fundamental and key step to thixoforming process^[8-9].

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It is well known that semi-solid isothermal heat treatment is a method developed in the mid-1990s, which omits the special procedure to fabricate the semi-solid billet but fulfill the semi-solid non-dendritic microstructure during heating prior to thixoforming^[10]. In addition, the whole process of semi-solid isothermal heat treatment is simple and low in cost. Therefore, it has aroused wide attention^[11-12]. The as-cast microstructure has a great effect on semi-solid microstructure prepared by isothermal heat treatment. Meanwhile, it has been proven that equiaxed as-cast microstructure is desired to subsequent treatment^[13]. When the size of billet is large, the size of solid particles demonstrates extremely apparent distinction from edge to centre zones during semi-solid isothermal heat treatment process of magnesium alloys^[14]. Zr is one of the most important elements which can evidently refine the microstructure, improve the homogeneousness of magnesium alloys^[15]. It is also well known that the addition of rare earth elements to Mg alloys can be an effective way to refine grains and improve the mechanical properties of alloys at ambient and elevated temperatures^[16]. Samarium (Sm) as one of the most economical RE elements with a large solubility in magnesium attracts significant interest^[17]. Therefore, it is rational to speculate that the addition of Sm possibly plays a beneficial role in the grain refinement of Mg-Zn-Zr alloys. Although the existing investigations have focused on the effects of Sm addition on the formation of the related phases and the mechanical properties, there have not been many investigations in the previous reports on the effect of Sm additions on the microstructural evolution of Mg-Zn-Zr alloy during partial remelting.

Therefore, the aim of the paper is to study the effects of Sm addition on microstructural evolution of Mg-6Zn-0.4Zr alloy in the process of semi-solid isothermal heat treatment. In addition, the investigation is conducted to obtain ideal semi-solid microstructures and to provide experimental basis and practical guidance for the future semi-solid forming.

1 Experimental procedure

Billets with nominal alloy compositions of Mg-6Zn-0.4Zr and Mg-6Zn-4Sm-0.4Zr were prepared from pure Mg, Zn ingot (99.9wt.%), Mg-30%Sm and Mg-25%Zr master alloys. All the raw alloys were preheated to 200 °C, and then melted in an electric resistance furnace with a mild steel crucible under the protective Ar atmosphere and a covering agent RJ-2. After pure Mg ingot was completely melted, the preheated Zn ingot was added to the melt at 680 °C. When the melt temperature increased to 760 °C, the Mg-Sm and Mg-Zr alloys were added to the melt and held for 20 min. Subsequently, 2wt.% (to the total mass of raw metals) C_2Cl_6 was added to the melt by mechanical stirring for scumming and deslagging at 740 °C, and then the melt was held for 10–15 min. At 730 °C, the melt was poured into a metallic mould preheated to 200 °C. The obtained ingots with the dimension of $\Phi 20\text{ mm} \times 180\text{ mm}$ were cut into cylindrical specimens of 20 mm in diameter and 10 mm in length for the isothermal heat treatment. RJ-2 was used

for protecting specimen from oxidation during isothermal heat treatment. In order to illuminate the effects of Sm addition and holding temperature on microstructural evolution during the heat treatment, the specimens were placed in the iron crucible and treated from 570 °C to 610 °C for 30 min, then all the specimens were rapidly quenched in cold water, and finally they were mechanically polished and etched in an 8% HNO_3 solution. The microstructural evolution was observed by using a MeF-3 optical microscope (OM) and the quantitative analysis of the alloy elements was carried out utilizing a JSM-6700F scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS). Phase constitutes were confirmed by a D/max-2400 type X-ray diffraction (XRD) unit using a Cu K α radiation with a scanning velocity of 5 °·min⁻¹ and a scanning angle from 10 to 90°. To quantitatively examine the size (d_0) and shape factor (f_0) of solid particles as well as liquid phase fraction, the obtained OM micrographs (at 100X magnification) were analyzed by Image Pro-Plus software according to the analytical formula^[5]:

$$d_0 = [\sum 2(A_0/\pi)^{1/2}]/n$$

$$f_0 = (\sum P_0^2/4\pi A_0)/n$$

where A_0 is the area of the solid particles, P_0 is the perimeter of each solid particle, n is the total particles account. If the shape factor (f_0) tends to be 1, the solid particles have a perfectly globular shape.

2 Results and discussion

2.1 As-cast microstructure

The SEM micrographs of the as-cast Mg-6Zn-0.4Zr and Mg-6Zn-4Sm-0.4Zr alloys are shown in Fig. 1. It is found that the microstructure of the experimental alloys consists of black α -Mg, gray eutectic phases distributed along grain boundaries and a few isolated particles inside the grains. The α -Mg grains demonstrate typical dendritic structure, the secondary dendritic arms of which have about equal length, and are separated by the eutectic phases. Compared with the microstructure of the Sm-free alloy, it can be clearly seen from Fig. 1(b) that the grains of the Sm-bearing alloy are obviously refined, and the length of the secondary dendritic arms becomes shorter, and meanwhile, the grain boundaries become extremely distinct. In addition, the eutectic morphology of the Sm-bearing alloy shows continuous net-work distribution while that of the Sm-free alloy presents disperse distribution at grain boundaries. The number of eutectic phases of the Sm-bearing alloy considerably increases. This is mainly attributed to the following reasons: On one hand, the addition of Sm results in Sm enrichment in front of the solidification interface, then the enrichment Sm effectively prevents the other alloy elements from diffusing to the matrix α -Mg, resulting in significant increase in quantity of the eutectic phases. On the other hand, the initially formed Sm-bearing intermetallics with high melting point disperse predominantly at the grain boundaries and cause an intensive constitutional undercooling in a diffusion layer ahead of the

Fig. 1: SEM micrographs of as-cast Mg-6Zn-xSm-0.4Zr alloy: (a) $x=0$; (b) $x=4$

advancing solid/liquid interface, leading to the decrease of the secondary dendritic arms and then refining the grains^[18]. So, it can be concluded that the addition of Sm can effectively refine the grains. In order to precisely confirm the phase constitute of the experimental alloys, the specimens were analyzed by using XRD. Figure 2 shows that the Sm-free alloy mainly contains α -Mg phase and the intermetallic Mg_2Zn_3 phase, while the Sm-bearing alloy also contains the $Mg_{41}Sm_5$ and $MgZnSm$ phases beside the α -Mg and Mg_2Zn_3 phases. According to existing studies, the $Mg_{41}Sm_5$ phase has a body-center tetragonal crystal structure^[19], and the $MgZnSm$ phase has a hexagonal structure^[20].

Fig. 2: XRD patterns of as-cast Mg-6Zn-xSm-0.4Zr alloy: (a) $x=0$; (b) $x=4$

2.2 Semi-solid microstructures at different zones

The optical micrographs of Mg-6Zn-xSm-0.4Zr alloy billet after isothermal heat treatment at 570 °C for 30 min are shown in Fig. 3. It can be seen that the original dendritic microstructures of the experimental alloys after heat treatment are changed into irregular and globular particles with some internally entrapped liquid phase. In addition, the solid particles of the Sm-free alloy exhibit distinctly dimensional effect, namely, the particles in the edge (with an average diameter of 172 μm) are smaller than those in the central zones (with an average diameter of 198 μm). Furthermore, the roundness of solid particles at the billet edge is better than that at the centre, and the liquid phase

fraction gradually decreases from the edge to the centre zones, as shown in Figs. 3(a) and (b). The dimensional distinction of solid particles is mainly resulted by the originally developed dendritic microstructure of Mg-6Zn-0.4Zr alloy. On the contrary, it is found from Figs. 3(c) and (d) that the solid particles of the Sm-bearing alloy billet have a smaller size, better roundness and nearly equal dimension in the whole zones except for a few larger solid particles in the centre of the alloy billet. It is well known that the transmission of heat has hysteretic nature characteristic. The existence of some larger solid particles is mainly due to the hysteretic nature of heat, leading to that the solid particles at the centre zones do not have adequate spheroidizing time. From the above experimental analysis, it can be concluded that the original microstructures have important effects on microstructural evolution of Mg-6Zn-0.4Zr alloy. That is to say, if the original microstructures of alloy billet are not refined, it is impossible to obtain perfect semi-solid particles during isothermal heat treatment.

2.3 Microstructural evolution at different holding temperatures

To further investigate the microstructural evolution of the Sm-free Mg-6Zn-0.4Zr alloy, the specimens were heated and held at different temperatures for 30 min. Figures 4 and 5 show the optical micrographs of non-dendritic microstructure, particle size, shape factor and liquid phase fraction, respectively, of the alloy treated at different temperatures for 30 min, respectively. All the microstructural evolution micrographs are taken from the centre of the billet. As seen in Fig. 4, with the increase of temperature, the dendritic microstructures completely disappear, while irregular and globular particles gradually form. It is found from Fig. 5 that the average size and shape factor of solid particles gradually decrease while the liquid phase fraction gradually increases with the increase of isothermal temperature. As shown in Fig. 4(a), at 570 °C, the eutectic phases with a low melting point begin to melt immediately. Then the α -Mg changes from developed dendrites to many irregular solid particles with non-uniform sizes. In addition, the percentage of solid phase is higher. Meanwhile, it can be seen from Fig. 5 that the average size of solid particles is 198 μm , the shape factor is 2.10 and liquid phase fraction is 8%, respectively. Both the

Fig. 3: Billet with alloy compositions of Mg-6Zn-xSm-0.4Zr alloy heat treated at 570 °C for 30 min: (a) and (b) $x=0$; (c) and (d) $x=4$

roundness of solid particles and the liquid phase fraction are relatively low. This is mainly because that lower isothermal temperature results in a very slow dendritic consolidation and dendrite-arm dissolution speed. Furthermore, some high melting point eutectic phases are not completely dissolved. With the temperature increasing from 580 °C to 600 °C, the liquid phase fraction gradually increases, while the particle size decreases little by little. Moreover, the shape of the solid particles has a tendency to become more globular, and the solid particles are entirely separated by liquid phase. The quantity of “liquid pool” within the solid particles increases gradually in this temperature range. Li Yuandong et al.^[11] found that during isothermal heat treatment process: within a grain, the homogenization and solubility of the alloying element can be improved with the increase of holding temperature, accelerating the evolution of non-dendritic microstructure. When the remelting temperature increases to 610 °C [Fig. 4(e)], all the solid particles are perfectly isolated by a great deal of liquid phase and become relatively round. In addition, the area of “liquid pool” obviously increases. The average size of solid particles

is 105 μm, the shape factor is 1.21 and liquid phase fraction is 44%, as illustrated in Fig. 5. It is well known that there is more liquid phase at a higher remelting temperature^[21]. The rapidly microstructural evolution is mainly attributed to the increased liquid phase, which makes the solid particles swiftly separate and thus promotes the microstructural evolution.

Figure 6 shows the optical micrographs of non-dendritic microstructure of Mg-6Zn-4Sm-0.4Zr alloy treated at different temperatures for 30 min, and Fig. 7 shows the particle size, shape factor and liquid phase fraction of the alloy. All the microstructural evolution micrographs were also taken from the centre of the billet. It can be seen from Fig. 6 that the non-dendritic microstructure consists of solid particles, liquid phase and “liquid pool” inside solid particles. As isothermal heat treatment proceeds, the eutectic phases are gradually dissolved, and meanwhile, the dendritic microstructures gradually change into irregular and globular particles through the consolidation of dendritic arms as well as the penetration and contagion of liquid phase. As shown in Fig. 6(a), at 570 °C, the microstructure is broken up to many finer, relatively round solid particles by

Fig. 4: Microstructure of Mg-6Zn-0.4Zr alloy heated at different temperatures for 30 min: (a) 570 °C; (b) 580 °C; (c) 590 °C; (d) 600 °C; (e) 610 °C

Fig. 5: Particle size, shape factor and liquid phase fraction of Mg-6Zn-0.4Zr alloy at different temperatures for 30 min

the liquid phase [compared with Fig. 4(a)]. In addition, the segmental solid particles still closely connect each other at some zones. It can be known from Fig. 7 that the average size of solid particles is 98 μm, the shape factor is 1.54 and liquid phase fraction is 15%, respectively. The corresponding microstructural variation is mainly attributed to the existence of residual eutectic phases in the interdendritic zones, leading to that many irregular solid particles are not completely separated. When the temperature varies from 580 °C to 600 °C, the roundness of solid particles tends to improve, and meanwhile, the number of solid particles gradually decreases while the distance between the solid particles and the liquid phase fraction gradually increase [compared with Fig. 6(a)]. The increase of liquid phase makes the microstructures change more rapidly from developed dendrites to irregular and globular solid particles^[22], as shown in Fig. 6(b) to 6(d). It can be known from Fig. 7 that both

the average size and shape factor of solid particles gradually decrease while the amount of liquid phase increases from 20% to 31%. It can be seen in Fig. 6(e) that, when the holding temperature exceeds 600 °C, all of the particles completely change into globular by the penetration of a great deal of liquid phase. Simultaneously, the liquid phase fraction increases sharply, and the finer and more rounded solid particles suspend in liquid phase [compared with Fig. 4(e) and Fig. 6(b) to 6(d)]. The average size of solid particles is 82 μm, the shape factor is 1.13 and liquid phase fraction is 43%, respectively.

Figure 8 shows the SEM micrograph of the Mg-6Zn-4Sm-0.4Zr alloy treated at 610 °C for 30 min. In order to analyze the atomic variation of Mg, Zn, Sm and Zr in “liquid pool”, inside solid particles and at the grain boundaries in the process of isothermal heat treatment, the compositions of the alloy at these locations are detected using EDS, and the results are listed

Fig. 6: Microstructure of Mg-6Zn-4Sm-0.4Zr alloy heated at different temperatures for 30 min: (a) 570 °C; (b) 580 °C; (c) 590 °C; (d) 600 °C; (e) 610 °C

Fig. 7: Particle size, shape factor and liquid phase fraction of Mg-6Zn-4Sm-0.4Zr alloy at different temperatures for 30 min

in Table 1. It is found that the contents of these elements are evidently different in “liquid pool” from inside the solid particle and at the grain boundaries. The contents of Zn and Sm in “liquid pool” (point A in Fig. 8) are obviously higher than those in solid particle and grain boundaries. This is attributed to the originally isolated eutectic particles, which are rich in Zn and Sm atoms during the initial solidification process. The atoms are reserved due to the relatively high solidification rate in the second solidification process. In addition, the contents of the Zn, Sm and Zr elements at grain boundaries (point C in Fig. 8) are significantly higher than that in the solid particle (point B in Fig. 8). This is mainly because that the second solidification rate is relatively high and that the Sm atom has a larger atomic radius than Zn and Zr, resulting in that the diffusion of Zn and Zr atom is restricted in the second solidification process, and these atoms concentrate at the grain boundaries. Thus, the residual levels of Zn and Zr atoms are relatively high at the grain boundaries.

Compared with the microstructural evolution of the Sm-free alloy, as the isothermal heat treatment temperature rises, the non-dendritic microstructure evolution of the Sm-bearing alloy is seemingly accelerated. That is to say, the desired globular particles with small dimension and good roundness are easily obtained from the Sm-containing alloy. In addition, the spheroidizing degree of solid particles is evidently improved. This can be explained by the following reasons. First, the coarse dendritic microstructures of the Sm-free alloy are refined by adding 4% Sm, and the finer the as-cast microstructures, the smaller and more globular the solid particles in the resulted semisolid microstructures^[23]. Second, the addition of 4% Sm promotes the creation of solid particles with some protuberances (Fig. 8) on the surface in the early stage of non-dendritic microstructure evolution. The protuberances on solid particle surface will lead to a decrease in melting point^[24]. It is well known that the surface curvature of solid particles can distinctly

Fig. 8: SEM image of Mg-6Zn-4Sm-0.4Zr alloy heated at 610 °C for 30 min

Table 1: EDS results of alloy heated at 610 °C for 30 min (at.%)

Location (point)	Mg	Zn	Sm	Zr
Liquid pool (A)	76.22	19.97	3.49	0.32
Inside solid particle (B)	98.65	0.78	0.35	0.22
Grain boundary (C)	81.21	15.91	2.26	0.62

influence its melting point, namely, the larger the curvature, the lower the melting point^[25]. As a result, the larger curvature solid particles are firstly melted, leading to optimized semi-solid microstructures. In addition, the variation of equilibrium melting point of the alloy is decided by the curvature of particles interface, which adheres the following equation^[26]:

$$\Delta T_r = -\frac{2\sigma k T_m V_s}{\Delta H_m} \quad (1)$$

where ΔT_r is the equilibrium melting point, σ is the interfacial tension between the solid and liquid phases, T_m is the melting point when the solid/liquid interface is plane, V_s is the molar volume of solid phase, k is the curvature of the solid/liquid interface, and ΔH_m is the Moore enthalpy of liquid-solid transformation. Therefore, according to equ. (1), the corners and edges of fine grains are preferentially melted so as to effectively promote microstructural evolution.

3 Conclusions

(1) The microstructure of as-cast Mg-6Zn-0.4Zr alloy consists of α -Mg and Mg_2Zn_3 phases. With 4% Sm addition, the corresponding microstructure is mainly composed of α -Mg, Mg_2Zn_3 , $Mg_{41}Sm_5$ and $MgZnSm$ phases.

(2) As compared with the Sm-free alloy, the eutectic morphology of the Sm-bearing alloy changes into continuous network. In addition, the size of solid particles evidently decreases while the number of eutectic phases distinctly increases by adding 4% Sm.

(3) The size of solid particles of the Sm-free billet with the

diameter of $\Phi 20$ mm exhibits a clear dimensional effect, namely, the solid particle sizes gradually increase from edge to center zones. Furthermore, the roundness of solid particles of the alloy billet tends to deteriorate from edge to the center zones.

(4) The apparent dimensional effect is basically eliminated, and the dendritic grains change to globular or elliptic particles by the addition of 4% Sm. For the Sm-bearing alloy, the distribution of solid particles tends to be homogenized, and meanwhile, the desired solid particles with small dimension and ideal globular shape can be easily obtained.

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