Microalloying Effect of Sn on Phase Transformation During Heat Treatment in Mg–Y–Zn–Zr Alloys

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Abstract

The microstructure and mechanical properties of the as-cast and heat-treated Mg–4.6Y–2.5Zn–0.6Zr– x Sn ($x = 0, 0.2$ and 0.5 wt%) alloys were investigated in this work. The results showed that the eutectics have been refined with 0.2% Sn addition and it has no effect on the phase category of the alloys. However, Sn_3Y_5 phase was found in 0.5% Sn-added alloy. After heat treatment at 520 °C, the transformation of the long-period stacking ordered (LPSO) phase takes place in the Mg–Y–Zn–Zr alloy, but the transition is not completed in the alloys containing Sn. In addition, during the heat treatment, the mechanical properties of Sn-free alloys are significantly improved, and the strength of alloys containing Sn does not change much. Through observation and analysis of the microstructure and mechanical properties, it is found that Sn addition hinders the process of α' -Mg $\rightarrow$ α -Mg + 14H and the process is the key to the transition of 18H-LPSO to 14H-LPSO.

Keywords Long-period stacking ordered (LPSO) phase · Microstructure · Heat treatment · Phase transformation

1 Introduction

Magnesium alloys with rare-earth (RE) elements have garnered considerable attention because of their excellent mechanical properties [1–4]. Mg–RE–Zn alloys are the most common alloys available because the long-period stacking ordered (LPSO) phase can significantly improve the mechanical properties of these alloys [5–7].

The LPSO phase in Mg–RE–Zn alloys features a multi-stacking fault structure [8]. Different stacking fault structures make different LPSO phase structures. Until now, six LPSO structures have been observed in Mg–RE–Zn alloys: 6H, 10H, 14H, 15R, 18R, and 24R [9]. As for the LPSO structures, the 18R and 14H structures are the major components [10–13]. The crystal structures of 18R and 14H remain controversial. It was suggested in recent works [12, 14] that 18R has an ordered base-centered monoclinic structure (lattice constants $a = 1.112$ nm, $b = 1.926$ nm, $c = 4.689$ nm, $\beta = 83.25^\circ$). Recent study by Zhu et al. [14]

has shown that the 18R phase has not only an ordered arrangement of its closely packed planes but also an ordered distribution of Y and Zn atoms within some of the closely packed planes. The composition of 18R is in fact Mg_{10}YZn instead of Mg_{12}YZn . Initially, it was reported that the 14H had a hexagonal lattice ($a = 0.321$ nm and $c = 3.694$ nm) [10]. However, recent studies [11, 14] indicated that the unit cell of 14H was, in fact, hexagonal with lattice parameters of $a = 1.11$ nm and $c = 3.65$ nm. The composition of 14H is Mg_{12}YZn .

Itoi et al. [15] first discovered that 18R LPSO phase transformed to 14H structure by annealing of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ (mol%) alloy at 773 K for 5 h and suggested that the 18R and 14H LPSO structures are phases at high temperature and low temperature, respectively. Abe et al. [16] reported that the construction of LPSO phases involves two processes, introducing stacking faults (SFs) in the α -Mg matrix and supplying the solute elements (Y and Zn) to the faulted layers. The stacking sequence of the common intrinsic stacking faults was ABABCACA [17], with the ABCA-type stacking sequence. From the stacking sequence point of view, 18R LPSO structure is constructed from several stacking faults, which can be easily formed around the interdendritic spacing and grain boundary, and the lamellar LPSO may nucleate through stacking faults. In addition, faulted 18R regions containing four α -Mg atomic layers

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provide a simple transitional path to 14H. During solution treatment at 773 K, the diffusion mobilities of Y and Zn atoms are accelerated and it is relatively easier for solute atoms to diffuse into the SFs since the faulted layers could be considered as high-diffusivity paths [18]. The diffusions of Y and Zn atoms into stacking faults will reach chemical ordering in order to form 14H structure. Once a 14H unit cell is nucleated, it could grow as time goes on [19]. Consequently, the formation of 14H LPSO structure is a diffusion-controlled process in this Mg–Y–Zn alloy.

However, the influence of the phase transition of LPSO on the stacking fault energy (SFE) during heat treatment is still not clear. Zhang et al. [20] calculated the stacking fault energy of Sn by a first-principles study and concluded that Sn could significantly lower the SFE of Mg. Decreasing the SFE leads to more stacking faults and forming more LPSO phases [21]. But there exist the intermetallic compounds Sn_3Y_5 and MgSnY with higher thermal stability, in equilibrium with the α -Mg matrix in the Mg–Sn–Y ternary system [22, 23]. In order to avoid the formation of impurity phases in Mg–Y–Zn, an experimental scheme of adding a small amount of Sn was used.

2 Experimental

The alloy ingots were prepared in an electromagnetic induction furnace using a mild steel crucible under Ar atmosphere. The raw materials were commercially available pure Mg, pure Zn, pure Sn, Mg–30 wt% Y, and Mg–30 wt% Zr. Three experimental alloys with the nominal compositions are given in Table 1. Samples were taken from the ingots. The Mg–4.6Y–2.5Zn–0.6Zr alloy and the Sn-modified alloys were solution treated at 520 °C for 8 h and then water quenched.

Samples of alloys for observation were etched in a solution of 10 ml water + 5 ml acetic acid + 25 ml ethanol after mechanical polishing. Then, the microstructures were characterized by optical micro-scope (OM), scanning electron microscope (SEM) with energy-dispersive spectroscopy (EDS) and transmission electron microscope (TEM). The phase constituents were identified by a D/max-2400 X-ray diffraction (XRD) using a CuK_α radiation with a scanning velocity of 5°/min and a scanning

angle from 20° to 80°. The tensile samples with a length of 80 mm and a center diameter of 5 mm were machined from the center of the alloy ingots. Tensile properties were determined from a complete stress–strain curve. 0.2% yield strength (YS), ultimate tensile strength (UTS) and elongation to failure (elongation) were obtained based on the average of three tests.

3 Results

3.1 As-Cast Microstructures

The optical micrographs of the as-cast Mg–4.6Y–2.5Zn–0.6Zr alloy with various Sn contents are shown in Fig. 1a–c. It can be clearly seen that the as-cast microstructure of Mg–4.6Y–2.5Zn–0.6Zr alloy has typical petaloid structure which consists of α -Mg matrix and coarse branching second phase (Fig. 1a). The second phase of alloys containing Sn becomes refined, and the secondary dendrite arm spacing (SDAS) decreases considerably with the increase in Sn contents (Fig. 2). The α -Mg turns into river-like morphology, and the interval between the second phase decreases considerably with increasing the Sn addition (Fig. 1b, c). The XRD patterns of the alloys in as-cast condition are given in Fig. 3a. As shown in Fig. 3a, the as-cast Mg–4.6Y–2.5Zn–0.6Zr alloy contains α -Mg, Mg_{10}YZn (18R-LPSO) and $\text{Mg}_3\text{Y}_2\text{Zn}_3$ (W). No secondary phase containing Sn could be observed in XRD.

To uncover more information regarding the secondary phase of the as-cast alloy, SEM was used to characterize the Mg–Y–Zn–Zr–xSn alloy (Fig. 4). Most of the eutectic phases are dendritic, with a small amount of granular eutectic phases distributed around them. Furthermore, it can be seen from Fig. 4b that the eutectics have been refined with 0.2% Sn addition. However, when the Sn addition is up to 0.5%, the eutectics tend to grow with the coarse and continuous morphology (Fig. 4c).

Figure 4a and Table 2 show that the eutectic phase in the Mg–4.6Y–2.5Zn–0.6Zr alloy is the gray LPSO and a very small amount of the white W phase. It is in line with the previous study. It can be seen from Fig. 4b and Table 2 that the 0.2% Sn-added alloy detects more the white W phase. Of course, there is still more the gray LPSO in the 0.2% Sn-added alloy. When the Sn addition is up to 0.5%, LPSO and W phase can be detected and their form has changed. However, elemental aggregation of Sn aggregate was found in 0.5% Sn-added alloy (Fig. 4c). EDS results (Table 2) show that the block phase consists of Mg, Y, and Sn. The atomic ratio of Y and Sn is approximately 5/3.

Table 1 Chemical compositions of studied alloys

Alloy	Composition (wt%)
A	Mg–4.6Y–2.5Zn–0.6Zr
B	Mg–4.6Y–2.5Zn–0.6Zr–0.2Sn
C	Mg–4.6Y–2.5Zn–0.6Zr–0.5Sn

Fig. 1 Optical microstructures of **a–c** as-cast and **d–f** solid solution treated **a, d** Mg–4.6Y–2.5Zn–0.6Zr, **b, e** Mg–4.6Y–2.5Zn–0.6Zr–0.2Sn, **c, f** Mg–4.6Y–2.5Zn–0.6Zr–0.5Sn alloys

3.2 Heat-Treated Microstructures

Figure 1d–f shows the optical micrographs of the alloys after solution treatment. It can be seen from Fig. 1d that the Mg–4.6Y–2.5Zn–0.6Zr alloy still has more secondary phases after heat treatment. After 8-h solution treatment at 520 °C, the morphology of the LPSO changes, being rod-shaped and flake-like, and is incorporated into a small part of the matrix. With the addition of Sn, the amount of the second phase of the alloys decreases dramatically, and most of the second phase is incorporated into the matrix. Only a small amount of granular second phase can be observed in the alloy with 0.2% Sn addition (Fig. 1e). When the Sn addition is up to 0.5%, the lamellar phase, the

small particle phase, and the massive phase can be observed in Fig. 1f. XRD patterns (Fig. 3b) of the alloys after solution show the LPSO phase decreases in the alloy with Sn addition after heat treatment. However, no peak of Sn has been observed in XRD yet.

The SEM images of the alloys with solution are shown in Fig. 4d–f. It can be seen from Fig. 4d that the LPSO presents as rod or sheet after heat treatment. The W phase is not found in the alloy. In the Mg–Y–Zn alloy, the 18R-LPSO to 14H-LPSO transition occurs at 520 °C. The existence of 14H-LPSO leads to good mechanical properties of the alloy. In Fig. 4e, the small particle phase and the thin rod phase are found in the Mg–4.6Y–2.5Zn–0.6Zr–0.2Sn alloy and the EDS results (Table 2) show that the

Fig. 2 SDAS of as-cast alloys

small particle phase is the W phase and the thin rod phase is Mg_{24}Y_5 . The LPSO is not found in the alloy after solution. In general, the LPSO will not be incorporated into the matrix during the heat treatment process [24]. It might be related to the redistribution of solute, which will also be discussed later. In Fig. 4f, the composition of the phases is more complex in the $\text{Mg-4.6Y-2.5Zn-0.6Zr-0.5Sn}$ alloy. The bulk phase, rod phase, and small phase can all be observed. The rod phase is proved to be LPSO by EDS (Table 2). In addition, it is worth mentioning that the bulk phase is aggregation of Sn and its composition is consistent

with that of the as-cast alloy. It shows that the bulk phase is a high-temperature stable phase.

Figure 5 shows the TEM image and corresponding SAED pattern of the 14H-LPSO phase in alloy A after heat treatment at 520 °C. It indicates that 14H is a more stable phase in alloy A after heat treatment at 520 °C. Then, alloy B is detected by TEM. But the 14H-LPSO phase is not found in alloy B. It is consistent with the results detected by SEM.

Figure 6 shows optical microstructure of alloy C after heat treatment at 520 °C for 20 h. It can be seen that the lamellar phase disappears and only a small amount of particles and a bulk phase are present. The microstructure of alloy C is similar to the microstructure of alloy B. It illustrates that the addition of Sn element has a significant effect on the formation of the LPSO phase in the alloy system.

3.3 Mechanical Properties

The yield strength, ultimate strength, and elongation of the alloys in different states are listed in Table 3. The typical stress–strain curves of the $\text{Mg-4.6Y-2.5Zn-0.6Zr-xSn}$ alloys in different states tested at room temperature are shown in Fig. 7.

With the addition of Sn, the yield strength and ultimate strength first increase and then decrease, and the elongation decreases in the as-cast alloys. As for the solution treatment, the introduction of Sn is not a good choice. It will lead to a decrease in performance.

Fig. 3 XRD patterns of **a** as-cast and **b** solid solution-treated alloys

Fig. 4 SEM micrographs of **a–c** as-cast and **d–f** solid solution treated **a, d** Mg–4.6Y–2.5Zn–0.6Zr, **b, e** Mg–4.6Y–2.5Zn–0.6Zr–0.2Sn, **c, f** Mg–4.6Y–2.5Zn–0.6Zr–0.5Sn alloys

4 Discussion

4.1 Effect of Trace Sn on Microstructure of As-Cast Alloys

Zhao et al. [25] investigated the Mg–1.0Sn– x Y ($x = 1.5\%$, 3.0%, 3.5, at.%) alloys, and it was found that only MgSnY phase is detected in the Mg–Sn–1.5% Y alloy, and the Sn_3Y_5 phase in the Mg–Sn–3.5% Y alloy, whereas both of them simultaneously exist in the Mg–Sn–3.0%Y alloy. The particle shape of MgSnY and Sn_3Y_5 phase, inherited from the solidification, has little change before and after hot extrusion. This is similar to the experimental results. Besides, the difference in electronegativity between the Y

atom and the Sn atom is larger than that of the Y atom and the Zn atom. It means that Y and Sn are more likely to form a phase when Y, Zn, and Sn are present at the same time. So, with the addition of Sn, minor changes have occurred in the composition of the second phase of the alloy. The evolution process can be expressed as follows (Fig. 8): lots of LPSO + trace W phase $\rightarrow$ lots of LPSO + small amount of W phase $\rightarrow$ lots of LPSO + small amount of W phase + small amount of Sn_3Y_5 phase. It results in a change in the morphology of the alloy microstructure.

As Ali et al. reported [26], Sn does not have the obvious effect on the grain refinement. However, the second phase of the alloys containing Sn becomes refined and the secondary dendrite arm spacing (SDAS) decreases

Table 2 EDS analysis (at.%) of typical positions in Fig. 4

Location (point)	Mg	Y	Zn	Zr	Sn
Figure 4a, A	87.99	4.31	7.30	0.41	–
Figure 4a, B	91.34	4.01	4.27	0.39	–
Figure 4b, A	92.88	3.20	3.49	0.38	0.08
Figure 4b, B	84.45	5.34	9.79	0.34	0.08
Figure 4c, A	93.95	2.78	2.86	0.32	0.09
Figure 4c, B	91.92	3.27	4.25	0.43	0.14
Figure 4c, C	47.95	28.62	1.78	3.26	18.39
Figure 4d, A	90.72	4.43	4.49	0.36	–
Figure 4e, A	83.35	14.14	1.53	0.80	0.19
Figure 4e, B	79.28	6.94	13.15	0.48	0.16
Figure 4f, A	92.63	3.24	3.77	0.25	0.11
Figure 4f, B	91.88	3.34	4.43	0.39	0.06
Figure 4f, C	60.23	14.04	5.00	9.45	11.48

Fig. 5 Bright-field TEM image and corresponding SAED pattern of 14H-LPSO phase in alloy A after heat treatment at 520 °C **Fig. 6** Optical microstructure of alloy C after heat treatment at 520 °C for 20 h

considerably with increasing the Sn addition as shown in Fig. 1b, c, which implies that the Sn content has an important effect on the SDAS of the investigated alloys. The mutative trend is considered to be mainly related to the following aspects. The SDAS can be calculated by the following equations [27]:

$$\lambda_2 = 5.5(Mt_f)^{\frac{1}{3}}, \quad (1)$$

$$M = \frac{\Gamma D \ln\left(\frac{C_s^m}{C_0}\right)}{m(1-k)(C_0 - C_1^m)}, \quad (2)$$

$$k = \frac{C_s}{C_1}, \quad (3)$$

where λ_2 is the SDAS, μm ; t_f is the local solidification time, s; M is the coarsening coefficient; Γ is the Gibbs–Thomson coefficient; D is the diffusion coefficient in liquid, m^2/s ; m is the liquid slope, $\text{K}/\text{wt}\%$; C_0 is the initial alloy concentration, $\text{wt}\%$; C_1^m is the eutectic composition, $\text{wt}\%$; k is the distribution coefficient; C_s and C_1 are the equilibrium solubilities. According to Eq. (1), λ_2 is affected by M and t_f . According to the classical solidification theory, the as-cast grain sizes are rated to the degree of heterogeneous nucleation and the constitutional undercooling [28]. When Sn was added to the melt, as solidification process proceeds, Sn would be absorbed onto the liquid ahead of the growing interface at relatively rapid cooling rate. Whereafter, it changes both the solid–liquid interfacial energy and the surface energy of the crystal, leading to hinder the diffusion of Zn and Y, which would increase the value of C_1 , and then the distribution coefficient k decreased. At last, according to Eq. (2), the decrease in k also results in the reduction of M . Consequently, when Sn is added to the alloy, λ_2 decreases evidently, as compared to the Mg–4.6Y–2.5Zn–0.6Zr alloy. In addition, it is well accepted that the growth process of crystal nucleus needs the atoms turn into the interior of crystal continuously after crystal nucleus formation [29]. During solidification process, the concentration difference of Sn near the solid–liquid interface changes due to the Sn redistribution; then, the Sn in liquid phase will prevent the diffusion of Mg, Zn, and Y atoms and the growth of grains, which favors the formation of shorter the SDAS.

4.2 Effect of Trace Sn on Phase Transformation in Heat Treatment Process

Abe et al. [16] have suggested that the construction of LPSO structure involves two procedures, introducing stacking faults periodically and supplying solute elements to and adjacent to the faulted layers. Since LPSO is a stacking ordered structure, the existence of stacking faults becomes an essential requirement for formation of 14H

Table 3 Tensile properties of Mg–4.6Y–2.5Zn–0.6Zr–xSn alloys in different states tested at room temperature

State	Alloy	YS (MPa)	UTS (MPa)	Elongation (%)
As cast	A	107	197	12.9
	B	124	231	11.1
	C	123	206	8.6
As solution treated	A	130	237	13.9
	B	125	230	13.4
	C	126	221	10.7

Fig. 7 Typical stress–strain curves of **a** as-cast and **b** solid solution-treated Mg–4.6Y–2.5Sn–0.6Zr–xSn alloys tested at ambient temperature **Fig. 8** Evolution processes of phase with addition of Sn in as-cast alloys: **a** Alloy A, **b** Alloy B, **c** Alloy C

structure. Simultaneous addition of Y and Zn elements could decrease the stacking fault energy of α -Mg grain on $(0001)_\alpha$ basal plane [30]. 14H lamellas are nucleated within α -Mg independently on the basis of basal SFs. Once the 14H cores are nucleated, they begin to grow accompanying with the diffusion of Y and Zn elements. For 14H phase, its growth involves two processes, broadening vertical to $(0001)_\alpha$ basal plane and lengthening along the basal plane [31]. Therefore, the broadening growth of 14H phase is an interface-controlled process, which is dominated by the

formation rate of ledges. Once a ledge is formed on basal planes, lengthening growth of 14H activates with the diffusion of Y and Zn elements.

Kim et al. [32] have presented a systematic atomic scale analysis of the structural evolution of LPSO structures. It suggested that the most probable transformation sequence of LPSO precipitate plates in the α -Mg matrix is as follows: single building block $\rightarrow$ various metastable LPSO building block clusters $\rightarrow$ 14H, and the most probable transformation sequence in the interdendritic LPSO phase is:

18R $\rightarrow$ various metastable LPSO building block clusters $\rightarrow$ 14H. And Liu et al. also got a similar conclusion in the experiment [31]. They suggested that the formation of 14H phase is not transformed from 18R phase, and its formation mechanism could be explained as α -Mg + 18R $\rightarrow$ α' -Mg $\rightarrow$ α -Mg + 14H. In this experiment, 14H phase does not appear in the alloys with Sn addition after heat treatment. They speculated that there are at least two possible reasons for this phenomenon: (1) Sn element changes the stacking fault energy of magnesium alloys, which prevents the 14H nucleation. (2) The addition of Sn reduces the stability of 14H phase, which hinders the conversion of the metastable phase to the stable phase.

To examine the stability of each LPSO structure, the enthalpy of formation with respect to unary hexagonal close-packed (hcp) Mg, Y, and Zn phases was evaluated according to the following equation [32]:

$$E_f(\text{Mg}_a\text{Y}_b\text{Zn}_c) = \frac{E(\text{Mg}_a\text{Y}_b\text{Zn}_c) - aE(\text{Mg}) - bE(\text{Y}) - cE(\text{Zn})}{a + b + c}, \quad (4)$$

where $E(\text{Mg}_a\text{Y}_b\text{Zn}_c)$ is the total energy of an LPSO structure and $E(\text{Mg})$, $E(\text{Y})$, and $E(\text{Zn})$ are the total energies of the unary phases. The thermodynamically most stable structures in both the α -Mg matrix and the interdendritic LPSO phase are a mixture of 14H and α -Mg. As mentioned in Sect. 4.1, Sn can change both the solid–liquid interfacial energy and the surface energy of the crystal, leading to hindering the diffusion of Zn and Y. On the other hand, the appearance of Sn_3Y_5 takes up a small amount of $E(\text{Y})$, which makes the formation of 14H more difficult. So, the appearance of such an intermediate phase is still possible. In addition, the solution-treated alloys are aged at 220 °C for 30 h and it is found that there is no obvious aging enhancement effect. It means that the disappearance of LPSO is not a simple dissolution of the matrix. Combining the above, it is speculated that Sn changes energy distribution of alloys and it may hinder the process of α' -Mg $\rightarrow$ α -Mg + 14H.

4.3 Effect of Trace Sn on Mechanical Properties of Alloys

In the as-cast alloys, with the addition of trace amounts of Sn, the strength of the alloys first increases and then decreases, and the elongation decreases. This may be affected by two factors. On the one hand, the addition of Sn makes the second phase more refined. At the same time, the second phase is more evenly distributed. The more fine and dispersed strengthening phase is the main reason for the increase in alloy strength [33]. On the other hand, the addition of Sn causes the increase in the W phase and the

appearance of Sn_3Y_5 phase. Both the W phase and the Sn_3Y_5 phase are brittle phases and they reduce the strength, and they also reduce the elongation. Experimental results show that adding 0.2% Sn can increase the strength of the alloy.

Comparing various alloys before and after heat treatment, the alloy without Sn has increased the strength and there is no change in the strength of the Sn-added alloys. This shows that 14H-LPSO has stronger enhancement than 18R-LPSO, and 18R-LPSO has similar enhancement effect to α' -Mg. The increase in elongation is due to the homogenization of the heat treatment.

5 Conclusions

1. The second phase of as-cast Mg–RE–Zn–Zr alloys containing Sn becomes refined, and the SDAS decreases with increasing the Sn addition. Adding 0.2% Sn to the alloy does not introduce new phases. But, when the Sn addition is up to 0.5%, the Sn_3Y_5 phase can be found in 0.5% Sn-added alloy.
2. After heat treatment at 520 °C, the transition of 18H-LPSO to 14H-LPSO takes place in Sn-free alloy. But the LPSO disappears or there is a small amount of 18R-LPSO in Sn-containing alloys. It may be that Sn addition hinders the conversion of 18R-LPSO to 14H-LPSO during heat treatment at 520 °C.
3. With the addition of Sn, the yield strength and ultimate strength first increase and then decrease, and the elongation decreases in the as-cast alloys. After heat treatment at 520 °C, the strength of alloys containing Sn does not change much, but the microstructure has changed a lot. It shows that Sn addition hinders the process of α' -Mg $\rightarrow$ α -Mg + 14H and the process is the key to the transition of 18H-LPSO to 14H-LPSO.

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