Effects of heat treatment on microstructure and mechanical properties of Mg-6Zn-4Sm-0.4Zr alloy

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Abstract: A new rare earth magnesium alloy (Mg-6Zn-4Sm-0.4Zr, wt.%) was prepared by permanent mould casting. The microstructure and mechanical properties of the alloy sample in as-cast and various heat treatment situations were characterized with an optical microscope (OM), X-ray diffractometer (XRD), scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS), transmission electron microscope (TEM) and mechanical tests at room temperature, respectively. The experimental results show that the as-cast alloy mainly consists of α -Mg, eutectic Mg_2Zn_3 , $MgZnSm$ and $Mg_{41}Sm_5$. These eutectic phases with continuous or semi-continuous morphology principally distribute along grain boundaries. Almost all the eutectic compounds dissolve in α -Mg and the grains have no obvious growth trend after optimum solution treatment at 490 °C for 18 h. Meanwhile, the ultimate tensile strength (UTS) of 229 MPa and elongation (EL) to rupture of 9.78% can be achieved through the optimal solution treatment, which increase by 37 MPa and 57.74%, respectively, compared with that of the as-cast alloy. Further aging treatments at 200 °C for different durations lead to the conspicuous increment of mechanical properties and prominent age-hardening response. Peak-aged alloy (treated at 200 °C for 12 h) reveals better mechanical properties (UTS 258 MPa, EL 9.42%, hardness 73.4 HV) compared with the same alloy treated in other aging conditions, which is mainly ascribed to precipitated Mg_2Zn_3 and $MgZn_2$ phases. Fracture analysis demonstrates that the as-cast alloy belongs to inter-granular and cleavage fracture patterns, while the solutionized alloy (treated at 490 °C for 18 h) reveals trans-granular and quasi-cleavage fracture modes. For the peak-aged alloy, the fracture pattern obeys the mixture of trans-granular and cleavage modes.

Key words: Mg-6Zn-4Sm-0.4Zr alloy; heat treatment; microstructure evolution; mechanical properties; fracture mode

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Magnesium alloys have many intrinsic advantages such as high specific strength and stiffness, excellent dimensional stability and damping, good castability and high recycle ability. These essential characteristics make them have great potential for applications in the fields of automobile, aerospace, electronic and telecommunication industries^[1-3]. Nonetheless, the utilization of magnesium alloys has been limited owing to their inherently inferior strength and poor formability^[4, 5]. In particular, it is known

that Mg-Zn binary alloys possess coarsely dendritic microstructure, wide freezing ranges and relatively poor mechanical properties^[6], and these drawbacks restrict the application of Mg-Zn binary alloys widely. Fortunately, it is well established that the mechanical properties of magnesium alloys can be improved via appropriate microalloying due to grain refinement and microstructure modification^[7]. Accordingly, many practical methods have been carried out to develop the new Mg-Zn based alloys with favorable comprehensive mechanical properties, such as innovative processing technology, grain refinement, heat treatment and microalloying^[8]. Among these approaches, microalloying and heat treatment are featured for their promising ability to achieve better mechanical performance through the solution strengthening and precipitation strengthening. As reported in a precious literature^[9], the ultimate tensile strength (UTS) and

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elongation (EL) of 363 MPa and 5.8% of the Mg-8Gd-3Y-0.5Zr alloy were achieved with proper solution and aging treatment.

In recent years, magnesium alloys with rare earth (RE) have received widespread attentions for their good creep resistance and excellent mechanical properties^[10]. It is well known that RE elements with unique atomic electron and chemical property can purify melt, modify microstructure, improve mechanical properties and enhance corrosion resistance^[11]. For example, the RE elements such as Gd, Y, Nd and Sm can effectively improve the mechanical properties of magnesium alloys^[12]. Samarium (Sm) is one of the cheapest RE elements. Its maximum solubility of which in Mg at eutectic temperature 815 K is 5.8%^[13]. It has been demonstrated that the solution and precipitation strengthening effects of Sm are higher than that of Nd in Mg alloys^[14]. The characteristics above have led to reascent interests on the development of the Sm-containing magnesium alloys. For example, Guan et al.^[15] found that the grains of the as-cast ZK60 alloy were refined and the dominant intermetallic phase was changed from MgZn₂ into Mg₃Sm₂Zn₃ phase by adding 1.5% Sm. Zheng et al.^[16] researched the microstructure, mechanical properties and creep behavior of Mg-3Sm-0.5Zn-0.4Zr alloy, where the UTS of 189 MPa, yield strength (YS) of 94 MPa and elongation (EL) of 11.7% were obtained. It is further reported that a UTS (215 MPa), YS (142 MPa) and EL (4.1%) of AZ61 alloy with 1.0% Sm after solution treatment were achieved^[17].

It is worth mentioning that, the majority of the magnesium alloy products are prepared by casting, and the high quality casting with fewer defects and homogeneous microstructure is desirable for the final service. An appropriate heat treatment technique should correspond to the uniform microstructure, highest strength, elongation, and hardness values. Solution treatment is an extremely effective method for eliminating or diminishing the destructive drawbacks of alloy. Meanwhile, subsequent aging treatment has a further prominent effect on the microstructure and mechanical properties of alloy. To promote the further development of the magnesium alloys, this work is aim to investigate the effects of heat treatment on the microstructural evolution and mechanical properties of Mg-6Zn-4Sm-0.4Zr alloy at room temperature. In addition, the obtained results can provide an experimental guide for the investigation of a new type of magnesium alloy.

1 Experimental procedures

The experimental alloy with nominal composition of Mg-6Zn-4Sm-0.4Zr (all the compositions are given in wt.% unless otherwise noted) was prepared from commercial pure Mg, Zn ingot (>99.99%), Mg-30%Sm and Mg-25%Zr master alloys. Firstly, all the raw materials were preheated to 200°C in an incubator, and then Mg ingot was melted in an electric resistance furnace with a mild steel crucible under Ar-shield. In addition, a covering agent RJ-2 was used to protect the melt from oxidation and burning in the whole process of melting. The matrix alloy melted completely at 650 °C, then raised the

temperature to 680 °C and added the Zn ingot into the melt. After the melt temperature was risen to 760 °C, the Mg-Sm and Mg-Zr alloys were added into the melt and held at this temperature for 20 min. Subsequently, the melt was refined by adding 2% C₂Cl₆ (ratio to the whole raw metal) to get rid of scumming and deslagging at 740 °C, then cooled to 730 °C and kept for 30 min. Finally, the melt was poured into metallic moulds pre-heated to 210 °C. Cast slabs with a cross-section of 85 mm × 130 mm and a gauge length of 210 mm and ingots with a diameter of 15 mm and a length of 180 mm were obtained, respectively.

According to existing literature^[18], the solid solubility of Sm in α -Mg increases with the increasing temperatures: 0.4% (200 °C) – 0.8% (300 °C) – 1.8% (400 °C) – 4.3% (500 °C) – 5.7% (540 °C). In order to explore the suitable solution treatment technology of the investigated alloy, considering the overburning at 495 °C in the experiment, the temperature of solution treatment was designed at 490 °C according to the variation trend of the solid solubility of Sm with temperature above. The solution time was set as 6, 12, 18, 24 and 36 h to get an optimal one among. The alloy samples were treated in an electrical resistance furnace (± 1 °C), followed by water quenching. To prevent oxidation, the samples were covered with a thin layer of sulfur powder during solution treatment. Subsequently, the samples treated by the optimal solid solution treatment were aged at 200 °C further with different durations ranging from 6 to 48 h with a gradient of 12 h in an oil-bath, and then taken out to do air-cooling.

The as-cast, solutionized and peak-aged alloy specimens for microstructural observations were prepared by pre-polishing and polishing, and then etched by nitric acid ethanol solution (4% nitric acid and 96% ethanol) and acetic-picric solution (5 g picric acid, 5 g acetic acid and 100 mL ethyl alcohol), respectively. The microtopographies were observed using OM and SEM. For the TEM characterization sample of peak-aged alloy, a slice with a thickness of 0.7 mm was firstly prepared using a computer numerical-controlled wire-cutting machine. Then, the obtained slice was manually polished to a thickness of 70–80 μ m, and then cut into 3 mm diameter discs. Subsequently, the polished sample was ion-milled with Fischione for TEM (TECNAIG2) operating at an accelerating voltage of 200 kV.

The tensile specimens with a cross-section of 2 mm × 3 mm and a gauge length of 15 mm were machined from the bottom of the obtained slabs using a computer numerical-controlled wire-cutting machine, and the dimension of tensile sample was

Fig. 1: Schematic for dimension of tensile sample (unit: mm)

shown in Fig. 1. Mechanical tests were performed on a WDW-100D universal material testing machine with a constant loading velocity of $1 \text{ mm} \cdot \text{s}^{-1}$ at room temperature. UTS and EL were obtained based on the average value of three tested specimens. The Vickers hardness was measured using a HvT-1000A tester with a loading of 0.98 N for 15 s.

The phase constitutions of the alloy were confirmed by XRD (D/max-2400) using a Cu $K\alpha$ radiation with a scanning angle span from 10° to 90° and a scanning velocity of $5^\circ \cdot \text{min}^{-1}$. The transverse fracture sections of the tensile samples were also characterized utilizing SEM to verify the detailed fracture mechanisms of the alloy in different states.

2 Results and discussion

2.1 As-cast microstructure

The OM and SEM micrographs of the as-cast alloy are shown

in Fig. 2, and the corresponding EDS results are shown in Table 1, respectively. It is clearly found from Fig. 2(a) that the whole microstructural morphologies of the as-cast alloy is a typical non-equilibrium solidification structure, and some eutectic phases cluster in local regions (marked by elliptical circles). Overall, there are two different kinds of morphologies for the original α -Mg grains, i.e. the large dendritic grains, such as the one marked α_1 , and the small granular grains, such as the one marked α_2 . The typical non-equilibrium solidification structure is comprised of original α -Mg with unidentical grain sizes and multiple shapes, continuous or semi-continuous interdendritic net-like eutectic phases and some isolated eutectic compounds located in the grain, which is shown in Fig. 2(b). A high-magnification SEM image is shown in Fig. 2(c), and it was used to investigate the morphology and distribution of eutectic phases. As seen in Fig. 2(c), the morphologically inhomogeneous distribution and three main

Fig. 2: Micrographs of as-cast alloy: (a) optical microstructure; (b) SEM microstructure; (c) local magnification

Table 1: EDS results of as-cast alloy (at.%)

Point	Mg	Zn	Sm	Zr
A	85.78	10.36	3.85	0.01
B	73.22	25.85	0.90	0.03
C	93.53	5.20	1.27	0.00

types of precipitates are distinctly observed, i.e. a terrimus precipitate marked A, bright precipitate marked B and shallow black precipitate marked C. Meanwhile, it is noteworthy that extremely obvious lamellar formations (marked by elliptical circle) are shown on the surface of the eutectic phase in the local region. According to the investigation^[19], the lamellar structure has a negative effect on the mechanical properties. The EDS results (Table 1) reveal that the precipitate A mainly includes Mg, Zn and Sm, and the content of Sm is significantly higher than that of other precipitates (B and C). The bright precipitate B is rich in Zn. The shallow black precipitate C contains massive Mg atom (compared with A and B).

2.2 Microstructure of solutionized alloy

The OM images of the alloy treated at 490°C for different solution times are shown in Fig. 3. It can be clearly seen that both the grain sizes and the whole eutectic morphologies of the alloy are modified, compared with the as-cast alloy (Fig. 2a). In addition, the outlines of grain boundaries gradually change to be relatively distinct with the solution time increasing. After being treated at 490°C for 6 h, as shown in Fig. 3(a), it is found that eutectic compounds were massively dissolved into matrix α -Mg, and the distribution of eutectic phases become relatively homogeneous compared with that in the as-cast alloy, the microstructural morphologies of primary α -Mg has been completely transformed into equiaxed grains. Simultaneously, the extent of dendritic segregation decreases markedly, the distribution of the secondary phases has completely changed into isolated particle morphology and tends to be more sparse and dispersible. However, there are still some eutectic phases remaining at grain boundaries, it implies that longer time is needed for completing dissolution.

With the solution time increased to 12 h, as shown in Fig. 3(b), the amount of residual intermediate compounds

Fig. 3: Microstructural evolution of alloy solutionized at 490 °C for different times: (a) 6 h, (b) 12 h, (c) 18 h, (d) 24 h, (e) 36 h

precipitated at grain boundaries and inside grains decrease further. The grain size is similar to that of the alloy heated for 6 h.

With the solution time increased to 18 h, as shown in Fig. 3(c), the eutectic compounds decrease obviously, which means that these phases are dissolved into the matrix as much as possible without overburning after solution treatment at 490 °C for 18 h, and the grain size does not show growth trend compared with the alloy heated for 12 h.

The microstructures of solutionized alloy treated at 490 °C for 24 h and 36 h are shown in Figs. 3(d) and (e). It is observed that the amount of residual eutectic phases and the extent of dendritic segregation decrease further. However, the polygonal grains grow obviously and it is difficult to distinguish the variation between those two microstructures shown in Fig. 3(d) and (e). However, severe oxidation has taken place on the surface of the alloy samples. According to the experimental analysis above, it can be concluded that the optimal solution treatment of the alloy is treating at 490 °C for 18 h.

2.3 Mechanical properties

Figure 4 shows the variations of mechanical properties versus aging time for the alloy solution treated at 490 °C for 18 h at room temperature, including the UTS, EL and micro-hardness. As shown in Fig. 4, the mechanical properties reveal a fluctuant variation pattern, and every cure has its own peak value. Furthermore, for the alloy, the heat solution treatment has a profound effect on mechanical properties. As shown in Fig. 4(a), in the as-cast condition, the UTS and EL are 192 MPa and 6.2%, respectively. After being solution treated at 490 °C for 18 h, the UTS of the treated alloy is 229 MPa, which increases by 19.27%, and the EL of the treated alloy is 9.78%, which increases by 57.74%, compared with the as-cast alloy.

Compared with the as-cast alloy, the increase of tensile properties of the solutionized alloy is caused by the following reasons: firstly, the solution treatment is conducive to diffusion of eutectic phases in matrix α -Mg due to a rapid dissolution

Fig. 4: Effects of aging time on mechanical properties of alloy: (a) tensile properties, (b) micro-hardness

rate, and this can result in the solution strengthening. Furthermore, the difference of atomic radius and elastic modulus between solute atoms and the matrix leads to the formation of lattice distortion and the stress field, which hinders dislocation movement^[20]. At last, intermetallic compounds are known to be the main sites for stress concentration, and as this stress increasing, they are potential sites for crack initiation^[20]. In a word, the reduction of harmful eutectic phases and dendritic segregation, as well as the elimination of stress concentration are beneficial to improve the tensile properties.

After aging treatment, it can be seen that the UTS is noticeable superior to that of the as-cast and solutionized alloy while the EL is inferior to that of the solutionized alloy. As shown in Fig. 4(a), the UTS exhibits rapid increase trend, whilst the evolution of EL presents slow increase tendency during the first aging period (6–12 h). Peak values of EL (9.42%) and UTS (258 MPa), are obtained at 12 h. Subsequently, the UTS and EL continuously tend to decrease with further increasing aging time. Until the aging time

increase to 48 h, UTS and EL decrease to 242 MPa and 8.78%, respectively.

The results show that the variations of the tensile properties as a function of aging time closely correspond to the microstructural evolution. The change of tensile properties of the aged alloy can be mainly ascribed to the reasons as follow: in the process of aging treatment, the amount of secondary precipitates increases with the aging time increasing, which can considerably improve the mechanical properties by precipitate strengthening and grain boundary strengthening.

A high-magnification SEM and bright field (BF) TEM micrographs were performed to help better illuminate the subtle characteristics of precipitated phases of the peak-aged alloy (200 °C, 12 h), as shown from Fig. 5. It is found in Fig. 5(a) that, for the aged alloy, there exist two types precipitated phase: similar cube phase marked A and analogous cuboid phase marked B. Fig. 5(b) shows a bright field (BF) TEM image of the aged alloy. As seen from Fig. 5(b), square-like precipitates marked C and lath-like ones lie in the basal plane. It's worth noting that the morphology of square-like precipitate

Fig. 5: SEM and TEM micrographs of alloy aged at 200 °C for 12 h: (a) SEM microstructure, (b) TEM microstructure

is similar to that of the one marked A. These precipitates can provide enough barrier to hinder dislocation movement, leading to the increment of mechanical properties.

A longer aging time (>12 h) inevitably induces the weakening of aging kinetics^[18] which leads to the gradual decrease of the amount of the precipitated particles, and this has a detrimental effect on the tensile properties. As shown in Fig. 4(b), the Vickers hardness increases rapidly in the first stage (0–12 h), and then appears to decrease from peak value to 67.1 HV (48 h), which is also mainly due to the decreased precipitates. Obviously, the alloy aged at 12 h has a peak value of hardness (73.4 HV) with shorter time and is taken as an optimum aging process.

2.4 XRD analysis

Figure 6 displays the XRD patterns of the alloy within different conditions. As illustrated in Fig. 6(a), the main phases are α -Mg, Mg_2Zn_3 , $MgZnSm$ and $Mg_{41}Sm_5$ in the as-cast

Fig. 6: XRD patterns of investigated alloy: (a) as-cast, (b) solutionized at 490 °C for 18 h, (c) aged at 200 °C for 12 h

alloy. According to existing literature, the $\text{Mg}_{41}\text{Sm}_5$ possesses a body-centered tetragonal crystal^[15], the MgZnSm phase has a hexagonal structure^[21]. Some Zr-containing phases are not detected in the XRD patterns. For comparison, after solution treatment (490 °C, 18 h), as shown in Fig. 6(b), the diffraction peaks of the secondary phases are markedly decreased, which indicate that massive eutectic compounds were dissolved in the α -Mg, it is highly consistent with the microstructure evolution of alloy treated by solution treatment shown in Fig. 3(c). Accordingly, the process of solution at 490 °C for 18 h is regarded as the optimal solution treatment for the alloy.

As shown in Fig. 6(c), the main peaks for the peak-aged alloy are corresponding to α -Mg and Mg_2Zn_3 phases. Furthermore, additional diffraction peaks of MgZn_2 phase were detected. According to the report^[22], the MgZn_2 phase belongs to a base-centered monoclinic structure ($a = 2.596$ nm, $b = 1.428$ nm, $c = 0.524$ nm, $\gamma = 102.5^\circ$), which contributed to the age-hardening response of the alloy during the aging process.

According to the XRD pattern, and combining with the result in Table 1, the precipitate A mainly includes Mg, Zn and Sm in the microtopography of as-cast alloy shown in Fig. 2(c) and the precipitate A is inferred as $\text{Mg}_{41}\text{Sm}_5$ phase or MgZnSm phase. The bright area marked B in Fig. 2(c) where is rich in

Zn can be validated as Mg_2Zn_3 phase. The shallow black phase marked C in Fig. 2(c) contains massive Mg atom (comparing A and B) and it is regarded as matrix phase.

2.5 Fracture analysis

Generally speaking, cleavage fracture, quasi-cleavage fracture and inter-granular fracture are three main rupture patterns in magnesium alloys^[23]. Figure 7 shows the SEM fractographs of the as-cast, optimum solutionized and peak-aged alloy after tensile fracture at room temperature. It is found that, in the as-cast alloy (Fig. 7a), the fracture surfaces are primarily composed of indistinct tearing ridges and a few porosities. The porosity is mainly attributed to shrinkage cavities during the original nonequilibrium solidification thermodynamic condition. In addition, SASAKI T et al.^[24] thought that porosity generally nucleates at inclusions. Because of these porosities, the bonding strength between α -Mg grains and eutectic phases is feeble, and this can provide the opening sites for micro-crack. Based on the fracture characteristics and the obtained relatively low mechanical properties, the fracture behavior of the as-cast alloy is characterized by inter-granular and cleavage fracture, which is mainly ascribed to the dendritic segregation and some harmful eutectic phases.

Fig. 7: SEM micrographs of fracture surface of alloy: (a) as-cast, (b) solutionized at 490 °C for 18 h, (c) aged at 200 °C for 12 h

As shown in Fig. 7(b), for the solutionized alloy (490 °C, 18h), the fracture surface morphologies present obvious differences, i.e. a large number of extremely obvious cleavage planes with tearing ridges and more prosperous river pattern compared with that of the as-cast alloy. Furthermore, some dimples are found, which are in accordance with the superior EL. According to the existing report^[25], the dimples are caused by localized microvoid coalescence owing to grain boundary sliding or dislocation motion. So, the fracture mode of the solution-treated alloy is considered as the mixture of inter-granular and quasi-cleavage modes.

As seen from Fig. 7(c), compared with the solutionized alloy, for the peak-aged alloy at 200 °C, a number of larger cleavage facets and grain boundaries can be observed clearly. This suggests that the fracture mode of the aged alloy is a mixed mode of trans-granular and cleavage modes.

3 Conclusions

(1) There are main α -Mg, Mg_2Zn_3 , MgZnSm and $\text{Mg}_{41}\text{Sm}_5$ phase in the as-cast alloy. The eutectic phases with continuous or semi-continuous morphology mainly distribute along grain boundaries.

(2) After the alloy is solutionized at 490 °C for 18 h, the dendritic segregation is completely eliminated and the grains have no evident growth tendency, meanwhile, the UTS and EL increase from 192 MPa and 6.2% (in as-cast state) to 229 MPa and 9.78%, respectively. Therefore, being treated at 490 °C for 18 h for the alloy is regarded as the optimal solution treatment.

(3) For the peak-aged alloy (200 °C, 12 h), besides α -Mg, Mg_2Zn_3 and $\text{Mg}_{41}\text{Sm}_5$ phases, the MgZn_2 phase is found. Furthermore, the peak-aged alloy has the excellent mechanical properties, i.e., UTS, EL and hardness of 258 MPa, 9.42% and 73.4 HV are obtained, separately.

(4) The fracture pattern of the as-cast alloy is dominated by

inter-granular and cleavage fracture while the rupture behavior of the alloy treated at 490 °C for 18 h is the mixture of inter-granular and quasi-cleavage modes. For the peak-aged alloy, the fracture behavior belongs to the mixture of trans-granular and cleavage modes.

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