Effect of Gd addition on mechanical and microstructural properties of Mg-xGd-2.6Nd-0.5Zn-0.5Zr cast alloys

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Abstract: The Mg-xGd-2.6Nd-0.5Zn-0.5Zr (x=0, 3.0, 4.5 and 6.0, wt.%) alloys were prepared by gravity casting and then T6 treatment. Microstructures of the alloy were observed using optical microscopy and scanning electron microscopy. Results show that the as-cast alloys contained network Mg₃Gd phases, blocky and needle-like Mg₁₂(Nd, Gd) phases. The α (Zr) particle inclusions in the α (Mg) matrix are also observed. Content of the secondary phases decreases as Gd content increases. Tensile test results show that the tensile and yield strengths of all the alloys increase as Gd content increases under the as-cast and T6 conditions, but the elongation exhibits the opposite trend. The blocky Mg₁₂(Nd,Gd) phases appear and act as crack initiator and deteriorates the experimental alloys' ductility with the increase of Gd content, especially as Gd content increases to 6.0wt.%, so the Mg-6.0Gd-2.6Nd-0.5Zn-0.5Zr alloy has the lowest elongation value compared to the other alloys studied. After T6 treatment, the Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy exhibits the optimal mechanical properties both at room temperature and 250 °C.

Key words: Mg-xGd-Nd-Zn-Zr alloys; mechanical properties; microstructure; Gd addition

CLC numbers: TG146.22

Document code: A

Article ID: 1672-6421(2020)03-212-07

As high-strength heat-resistant magnesium alloys, Mg-RE (rare earth) alloys have an extensive application prospect in automotive and aerospace industries [1]. The Mg-Nd-Zn-Zr alloy has been used as a heat-resistant magnesium alloy, the ultimate tensile strength (UTS) was up to 230 MPa at 200 °C [2]. But the mechanical properties of Mg-Nd-Zn-Zr alloys are inadequate for the possible application at elevated temperature up to 250 °C due to the low thermal stability of precipitates and thereby a rapid decrease of mechanical properties with increasing temperatures [3]. Recently, Gd was found to be able to further improve the yield strength (YS) and to enhance the heat resistance of Mg-Nd-Zn-Zr alloy because of the fact that Gd can enhance thermal stability of second phase and even form

strengthening phase with a high thermal stability [4]. Gd has been widely used to add in Mg alloys [5,6]. The high solid solution of Gd in the Mg matrix plays an important role in strengthening of Mg alloys [7]. Generally, the dominant intermetallic phase in Mg-Gd-RE-Zr alloys is Mg₅RE. Nevertheless, Zn was frequently added into Mg-RE based alloys to enhance age hardening response [8-9]. The addition of Zn to Mg-RE alloys lowers the addition amount of RE while can achieve the similar mechanical properties [10]. Zr is an excellent grain refining element and can significantly enhance the mechanical properties of Mg alloys [7]. Gd and Nd were selected as the main alloying elements with microalloyed Zn and Zr. The addition of Gd and Nd could enhance tensile strength of the Mg [11]. The Mg-Nd-Gd-Zn-Zr alloys seem particularly attractive because they exhibit a stronger hardening response with lower overall RE levels [12]. Addition of 0.5wt.% Zn to Mg-3wt.%Nd alloy can further increase the peak-aged hardness of the alloy [13]. It has been reported that the addition of 0.6wt.% Gd in Mg-3.4Nd-0.1Zn-0.4Zr(wt.%) alloy can increase the thermal stability of Mg₁₂Nd phase and improve high temperature properties [14,15]. The UTS up to 215 MPa

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Received: 2019-12-17; Accepted: 2020-04-21

at 250 °C was obtained from the Mg-3.4Nd-0.1Zn-0.4Zr alloy. Adding 8wt.% Gd into Mg-3Nd-0.6Zr (wt.%) alloy was also found to promote the precipitation of a large amount of Mg₅Gd phase with a high thermal stability^[16]. The UTS of Mg-3Nd-8Gd-0.6Zr alloy reaches 222 MPa at 250 °C. Addition of 1.82wt.% Gd into the Mg-2.49Nd-0.19Zn-0.4Zr alloy resulted in an outstanding age-hardening because of the formation of rod-shaped metastable β'' and β' phases; and at 250 °C, the UTS up to 220 MPa was obtained^[17]. But, effects of Gd addition on microstructure and mechanical properties of Mg-Nd-Zn-Zr based alloys were rarely reported.

In this study, using the Mg-(2.0–3.0)Nd-(0.2–0.7)Zn-(0.4–1.0)Zr (ZM6) alloy as the matrix alloy, by addition of Gd, the new types of Mg-xGd-2.6Nd-0.5Zn-0.5Zr ($x=0, 3.0, 4.5$ and 6.0 , wt.%) alloys were designed. Extensive analysis was performed to study the microstructure and mechanical properties of these alloys.

1 Experimental procedure

Commercial pure Mg and Zn (99.99%) were melted in a 30 kg crucible in an electrical-resistance furnace under the protection of the anti-oxidizing flux, Nd, Gd and Zr were added as Mg-30%Nd, Mg-30%Gd and Mg-30%Zr alloys, respectively. Then, at 760 °C, the melt was poured into a steel mold pre-heated to 200 °C by gravity casting. Solid solution treatment of the alloy was performed using a hot-air circulation furnace with an accuracy of ± 5 °C, under the protection of FeS. Samples were solution-treated at 530 °C for 14 h, followed by quenching into

warm water of 60–80 °C. Aging treatment of the alloy was performed at 205 °C for 16 h, followed by air cooling (T6).

Hardness of the alloys was measured using a PH600 Brinell tester. Totally, five measurements were performed for each alloy; removing the minimum and maximum values, the average of the remaining three values was the final result. Tensile properties were obtained using a CSS-1120 tensile tester equipped with an electric resistance furnace containing a fully enclosed chamber capable of operating at 25–300 °C. The final result was the average of three measurements.

Microstructures of the alloys were analyzed by using of an Olympus PM-G3 scanning electron microscope (SEM) and a JEOL JSM-5800 optical microscope (OM). X-ray diffraction (XRD) was performed using an X'Pert Pro MPD instrument. Phase constitutions of the alloy were analyzed using Sirion 200 field emission scanning electron microscopy (FE-SEM) coupled with Oxford energy dispersive spectroscopy (EDS). Transmission electron microscopy (TEM) was performed using JEM2100F operated at 200 kV.

2 Results and discussion

2.1 Microstructure

2.1.1 As-cast microstructure

Microstructures of the as-cast Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys ($x=0, 3.0, 4.5$ and 6.0) are composed of almost equiaxed flower-shaped α (Mg) grains, as shown in Fig. 1. The semi-continuous eutectic compounds are unevenly distributed at the grain boundaries. A lot of needle-like precipitates with different

Fig. 1: Optical micrographs of as-cast Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys: (a) 0; (b) 3.0; (c) 4.5 and (d) 6.0

orientations and intermetallic particles are also observed within the $\alpha(\text{Mg})$ grains with an increasing amount as Gd content increases from 0wt.% to 4.5wt.% in Figs. 1a to 1c, and disappear as Gd content further increases to 6.0wt.% in Fig. 1d. Volume fraction of the laminar eutectic compounds distributed at the grain boundaries increases as Gd content increases from 0wt.% to 6.0wt.%. The grain sizes of the Mg-xGd-2.6Nd-0.5Zn-0.5Zr ($x=0, 3.0, 4.5$ and 6.0) alloys are about 28.3 μm , 23.8 μm , 20 μm and 20 μm , respectively. It can be seen that the grains of the Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys are refined after Gd increases from 0wt.% to 4.5wt.% (Fig. 1a–1c), but the grain size remains almost the same as Gd further increases to 6.0wt.%.

Figure 2 shows the SEM and EDS analyses of the as-cast Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys ($x=4.5$). There are lots of the needle-like phases (marked “A” in Fig. 2a) near the grain boundaries. Its composition was determined to be Mg-5.48Nd-

4.09Gd-1.22Zn (at.%) by EDS analysis (Fig. 2b), which is close to $\text{Mg}_{12}(\text{Nd,Gd})$ phase. EDS analysis of the minor phase appearing as white dots inside $\alpha(\text{Mg})$ grains (marked “B” in Fig. 2a) shows that it corresponds to $\alpha(\text{Zr})$ phase (Fig. 2c). This type of particles are frequently observed in Mg-RE-Zn systems and confirmed to be Zr-rich particles or Zn-Zr compounds^[18].

The network-shaped phase (marked “C” in Fig. 2a) was actually composed of network precipitates at higher magnification, as shown in Fig. 3a. These precipitates were confirmed to be Mg_3Gd with face centered cubic (FCC) structure (Fig. 3b). The chemical composition of the blocky phase (marked “D” in Figs. 2a) determined using EDS analysis is Mg-3.98Nd-3.25Gd-1.08Zn (Fig. 2d). These precipitates were confirmed to be $\text{Mg}_{12}(\text{Nd,Gd})$ with a tetragonal structure according to the SAED pattern, as shown in Fig. 4b. This result was also confirmed by XRD pattern, as shown in Fig. 5.

Fig. 2: SEM (a) and EDS (b) of the as-cast Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy

Fig. 3: TEM bright field image (a) and SAED pattern of network-shaped precipitates inside $\alpha(\text{Mg})$ matrix (b)

Fig. 4: High magnification TEM bright field image (a) and SAED pattern of the blocky precipitates formed inside the $\alpha(Mg)$ matrix (b)

Fig. 5: XRD of Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys ($x= 0, 3.0, 4.5$ and $6.0, wt.\%$)

2.1.2 Microstructure after T6 treatment

Figure 6 shows the microstructure evolution of Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys ($x = 0, 3.0, 4.5$ and $6.0, wt.\%$) after T6 treatment. It is found that the grain boundary becomes more obvious than the as-cast alloys. The clusters of precipitated phases are observed with some $\alpha(Mg)$ grains, the amount of these precipitates increases as Gd increases from 0 to 4.5wt.%, but decreases as Gd content is further increased from 4.5wt.% to 6.0wt.%, as shown in Fig. 6. The needle-like phase of the as-cast alloys disappears, but the block structure of about 0.5–3.0 μm forms at grain boundaries. They are unevenly distributed along the boundaries, and their overall amount increases as Gd content increases from 0 to 6.0wt.%.

SEM micrographs and EDS analysis of the T6 treated Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy are shown in Fig. 7. The magnified SEM images in Fig. 7b show that the precipitates

Fig. 6: Optical micrographs of T6 Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys: (a) $x=0$; (b) $x=3.0$; (c) $x=4.5$ and (d) $x=6.0$

Fig. 7: SEM micrographs of T6 treated Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy

consist of long needle-like phases (marked “A” in Fig. 7b) with different orientations and some long rod-like precipitates (marked “B” in Fig. 7b). Besides, the composition of the blocky-shaped phase at grain boundaries (Fig. 7c) is 91.39Mg-6.26Nd-1.54Gd-0.81Zn(at.%) by the EDS, which is close to the composition of a $Mg_{12}(Nd,Gd)$ alloy.

2.2 Mechanical properties

Figure 8 shows the hardness (HB), elongation (EL), ultimate tensile strength (UTS) and yield strength (YS) of the alloys under different states. All the HB, UTS, and YS increase continuously as Gd content increases from 3.0wt.% to 6.0wt.% under the as-cast and T6 conditions. However, the elongation shows the opposite trend. The elongation of the Mg-6.0Gd-2.6Nd-0.5Zn-0.5Zr alloy is only 3.0%, about 60% lower than the Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy after T6 treatment. So, the Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy after T6 treatment has the best comprehensive mechanical properties. The HB,

EL, UTS, and YS of the Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy after T6 treatment are 92 HB, 4.5%, 325 MPa, and 302 MPa, respectively. The alloy after T6 treatment even exhibits a much higher tensile strength and better ductility than WE43 alloy (containing 4.2wt.% of Y, 4.0wt.% of RE and 0.5wt.% of Zr) at 250 °C (Table 1).

2.3 Fracture behavior

Figures 9a–9c show the SEM tensile fractures of the Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys (x=3.0, 4.5 and 6.0) tested at room temperature after T6 treatment. Fracture surface of the alloys shows typical brittle fracture characteristics with numerous cleavage planes and micro-cracks. Gd has an obvious effect on the fracture behavior of the T6 treated Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys. Amount of the white cuboid-shaped residual phases increases with an increase in Gd content. The magnified of the blocky-shaped phase is shown in Fig. 9d. Composition of the white blocky-shaped

Fig. 8: Room temperature mechanical properties of Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys

Table 1: Tensile properties of T6 treated Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr and WE43^[12-13] alloys

Materials	YS (MPa)		UTS (MPa)		EL (%)	
	Room temp.	250 °C	Room temp.	250 °C	Room temp.	250 °C
Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr	205	145	325	245	4.5	18.5
WE43	178	155	250	211	7.0	18.0

Fig. 9: Tensile fracture of T6 treated Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys at room temperature: (a) $x=3.0$, (b) $x=4.5$, and (c) $x=6.0$, (d) magnified picture of blocky phase, (e) EDS analysis of blocky particles

Fig. 10: Tensile fracture of T6 treated Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys at 250 °C: (a) $x=3.0$; (b) $x=4.5$ and (c) $x=6.0$

residual phase determined using EDS is Mg-6.10Nd-1.22Gd-0.12Zn [Fig. 9(e)], which is a $Mg_{12}(Nd,Gd)$ based alloy. The $Mg_{12}(Nd,Gd)$ phase has a poor elongation, which is why the elongation of Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys worsens with Gd addition.

Fracture features of the Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys ($x=3.0, 4.5$ and 6.0) after T6 treatment followed by tensile tests at 250 °C (Figs. 10a–10c) show more dimples and tear ridges than the alloys tested at room temperature, indicating the high

elongations of these alloys. The elongation is 21.5% for the Mg-3.0Gd-2.6Nd-0.5Zn-0.5Zr alloy after T6 treatment.

3 Conclusions

Microstructure and mechanical properties of the as-cast and T6 treated Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys were investigated. Major results as follows can be obtained:

(1) Microstructures of the as-cast Mg-xGd-2.6Nd-0.5Zn-

0.5Zr alloys consist of equiaxed $\alpha(\text{Mg})$ grains, $\alpha(\text{Zr})$ particles and semi-continuous laminar eutectic compounds distributed along the grain boundaries. Volume fraction of these laminar eutectic compounds increases as Gd content is increased from 0 to 6.0wt.%. Numerous needle-shaped precipitates with different orientations as well as intermetallic particles are also detected inside the $\alpha(\text{Mg})$ grains of the alloys with 0wt.%–4.5wt.% Gd, but not found in the alloy with 6.0wt.% Gd.

(2) All the hardness, yield strength and ultimate tensile strength of the as-cast and T6 treated alloys increase continuously as Gd content increases from 3.0wt.% to 6.0wt.%, both at room temperature and 250 °C. However, the elongation is opposite. The optimal mechanical properties after T6 treatment are obtained for the Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy. The comprehensive mechanical properties of the Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr alloy at 250 °C are even better than those of commercial WE43 alloy. Thus, Mg-4.5Gd-2.6Nd-0.5Zn-0.5Zr is a very promising material with excellent mechanical properties and a significantly lower cost than commercial WE43 alloy.

(3) Addition of Gd also affects the fracture behavior of the experimental alloys after T6 treatment. Fracture surfaces of all the Mg-xGd-2.6Nd-0.5Zn-0.5Zr alloys ($x=3.0, 4.5$ and 6.0 , wt.%) show typical brittle fracture characteristics at room temperature. While, at 250 °C, more dimples and tear ridges are found, indicating the high elongations of these alloys at elevated temperature.

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