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Effect of homogenization on microstructure evolution and mechanical properties of aged Mg-Nd-Sm-Zn-Zr alloy

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Abstract

As an indispensable pre-treatment for aging, homogenization treatment has a significant effect on precipitation behavior of the Mg-RE alloys. Herein, the influence of homogenization temperature on the microstructure evolution and mechanical performance of a novel Mg-2.0Nd-2.0Sm-0.4Zn-0.4Zr (wt.%) alloy has been studied systematically. The results indicated that the as-cast alloy was mainly composed of $\alpha$-Mg matrix, $\beta$-Mg12(Nd,Sm,Zn) phase and Zr-containing particles. Upon increasing the homogenization temperature from 500 $^\circ$C to 525 $^\circ$C for 8 h, the average grain size of as-homogenized alloy increased from 76 $\mu$m to 156 $\mu$m, and the content of $\beta$ phase decreased gradually. It was worth noting that the homogenization temperature exceeded 515 $^\circ$C, the $\beta$ phase at the grain boundaries was completely dissolved. After aging at 200 $^\circ$C for 18 h, numerous of plate-like $\beta$ phases were observed in $\alpha$-Mg matrix. The rise in homogenization temperature was conducive to nucleation and growth of the $\beta'$ phase. However, excessive homogenization temperature significantly coarsened grain size. The aged alloy under homogenization treatment at 515 $^\circ$C for 8 h achieved optimal mechanical properties. The values of ultimate tensile strength, yield strength and elongation were 261 MPa, 154 MPa and 5.8%, respectively. The fracture mode of the aged alloy mainly exhibited a typical transgranular cleavage fracture.

1. Introduction

Magnesium (Mg) - rare earth (RE) alloys have been greatly developed in the past decades due to their low density, good corrosion resistance and potential for energy saving [1-5]. Hence, the Mg-RE alloys are widely applied in aerospace, electronic communication and automobile applications [6]. However, current commercial high strength Mg-RE alloys mostly contain a large amount of heavy RE elements, which increase costs and densities of the alloys. It is not conducive to energy saving and light weight [7]. Based on this, the Mg-RE alloys containing light RE element have gradually became a research hotspot in recent years. In particular, the novel Mg-Nd-Sm-Zn-Zr alloys have attracted much attention owing to its excellent corrosion resistance and biocompatibility [8]. However, considering that the strength of the as-cast Mg-Nd-Sm-Zn-Zr alloy is still insufficient as the structural material. Hence, the heat treatment is used to further enhance the performance of the as-cast alloy [9]. As indispensable pre-treatment for the aging, the homogenization temperature plays crucial role during the heat treatment [10]. The homogenization treatment is performed at elevated temperature, in this regard, the second phase completely dissolve. Hence, it is very important to control the parameters of homogenization treatment. Xu and Kamado et al [11] found that the eutectic $\beta$ phase of AZ91 alloy could dissolve effectively after
homogenization at 415 °C. As the homogenization time prolonged, the solute atoms uniformly distributed in the α-Mg matrix. However, compared with the homogenization time, the homogenization temperature had more obvious effects on the microstructure of the alloy. In addition, a higher concentration of quenched vacancies can be formed after the alloy quenched at a higher temperature. It was beneficial to enhance the diffusion rate of solute atoms, while nucleation and growth of precipitates were promoted significantly [12]. For example, Xu et al. [13] revealed that the number of the β phase was largely increased in the aged alloys due to more nucleation site of β phase and higher concentration of solute atoms achieved at higher homogenization temperature. However, the excessive homogenization temperature will lead to grain coarsening even overburning. Obviously, the mechanical properties of alloy could be deteriorated seriously [14]. Or vice versa, the inadequate homogenization temperature can reduce the response of aging strengthening [15]. It is not conducive to the full play of mechanical properties of the Mg-RE alloys. Therefore, study on homogenization temperature of the Mg-RE alloys in depth is very necessary to developed high performance Mg-RE alloys. Up to now, the studies on the homogenization treatment were mainly focused on the Mg alloys containing heavy RE element or the Mg alloys with mixed heavy and light RE element [16–18]. The research on the homogenization temperature of the Mg alloys only containing light RE element was inadequate.

Hence, current work was performed to explain the influence of homogenization temperature on the microstructure evolution and mechanical properties of aged Mg-2.0Nd-2.0Sm-0.4Zn-0.4Zr alloy. The mechanism of strengthening and fracture of the alloy were revealed. Meanwhile, the optimization of heat treatment parameters was also an important consideration for the commercial application of the novel Mg-Nd-Sm-Zn-Zr alloy.

2. Materials and methods

The nominal composition of the alloy is the Mg-2.0Nd-2.0Sm-0.4Zn-0.4Zr (wt.%). The experimental ingot was melted in an electric resistance furnace under a mixed SF6/CO2 with the volume ratio of 99:1 to avoid oxidation of melt. The pure Mg, Zn and all master alloys were preheated at 200 °C–300 °C for more than 3 h to dry. First, the pure Mg was added into a steel crucible at approximately 720 °C. After the pure Mg was completely melted, the Mg-30wt.%Nd alloy, Mg-25wt.%Sm alloy and pure Zn were added into the melt as the temperature raised to about 750 °C. Subsequently, the Mg-25wt.%Zr alloy was added into the melt when the temperature reached to about 780 °C. The melt was poured into a permanent mold at about 700 °C after stirring and slag removal, and the mold was preheated at 300 °C for 3 h. The dimension of mold was 150 mm × 120 mm × 30 mm. According to our previous work, the homogenization temperatures of as-cast alloy were determined to be 500 °C, 505 °C, 510 °C, 515 °C, 520 °C, and 525 °C, respectively. For avoiding the formation of quenching crack, all as-homogenized alloys were quenched in hot water of about 80 °C. And then, all as-quenched samples were aged at 200 °C for 18 h.

The samples for optical microscopy (OM) and scanning electron microscopy (SEM) were mechanically ground, polished, and subsequently etched by the Acetic-Picral solution. The average grain size of as-homogenized alloys was measured by the linear intercept method from at least five OM images under low magnification. The x-ray diffraction (XRD) and energy-dispersive x-ray spectroscopy (EDS) were used to analyze the chemical composition and type of phase. The aged samples for transmission electron microscopy (TEM) were milled under 4 kV ion gun energy. The tensile tests were carried out by MTS-E44 testing machine. The rectangular cross section of tensile samples was 2 mm × 5 mm, and the gauge length was 15 mm. The strain rate of the tensile test was 1 × 10−3 s−1. Each tensile condition was repeated for at least five times to achieve the reproducibility of results. The morphologies of tensile fracture were observed by SEM.

3. Results and discussion

3.1. The microstructure of as-cast alloy

The figure 1 shows the OM image and the XRD diffraction patterns of as-cast alloy. The microstructure of as-cast alloy mainly consisted of α-Mg matrix and continuous network-shaped β phase. The primary α-Mg phase formed firstly during the solidification, and the Mg element was consumed continually in the liquid metal. The content of rare earth elements and Mg element in residual liquid gradually reached eutectic point, and the products of eutectic reaction were α-Mg phase and eutectic phase. The diffraction peaks of the α-Mg matrix and the β phase could be seen in the XRD spectrogram, as shown in figure 1 (b). The irregular β phase was mainly Mg12Nd-type phase. In order to further determine chemical elements of the β phase, the SEM image and corresponding EDS results are shown in figure 2. The β phase and α-Mg matrix were marked by point A and point B, respectively. The EDS results of point A revealed that the β phase mainly composed of Mg element, Nd element, Sm element, and Zn element. Hence, according to the results of the XRD, the chemical composition of
the β phase could be identified as Mg12(Nd,Sm,Zn), which is a typical hard and brittle phase [19]. Furthermore, the EDS results of the point B exposed that in addition to Mg element, the α-Mg matrix also contained trace amounts of Nd element, Sm element, Zn element, and Zr element.

3.2. Effect of homogenization temperature on microstructure

The SEM images and corresponding the EDS results of the as-homogenized alloy under homogenization treatment at (a) 500 °C, (b) 505 °C, (c) 510 °C, (d) 515 °C, (e) 520 °C, and (f) 525 °C for 8 h are shown in figure 3. With increasing the homogenization temperature from 500 °C to 510 °C, the content of β phase decreased gradually, as shown in figures 3(a) to figure 3(c). More importantly, when the homogenization temperature exceeded 515 °C, the β phase had almost completely dissolved into the α-Mg matrix. Meanwhile, some Zr-containing particles can be observed within the grain. Corresponding the EDS results of the Zr-containing particles are shown in figures 3(d) to figure 3(f) (marked by the red line).

For further understanding the effect of homogenization temperature on the grain size of the as-homogenized alloys, the corresponding OM images are given in figure 4. Moreover, the average grain size of the
as-homogenized alloy is presented in figure 5. Clearly, the average grain size increased by 30.26% from 76 μm to 99 μm when the homogenization temperature raised from 500 °C to 515 °C. However, with the homogenization temperature further increased to 525 °C, the average grain size increased by 57.57% from 99 μm to 156 μm. It should be noted that the increment of average grain size was nearly double from 30.26% to 57.57%. The above experimental results indicated that the β phase at grain boundary had a strong inhibition on movement of grain boundaries during the homogenization process. Therefore, the average grain size increased obviously when the homogenization temperature exceeded 515 °C. The coarse grains could deteriorate the mechanical properties of the alloy. It is judged that the 515 °C was the appropriate homogenization temperature for the alloy.

In order to understand the effect of homogenization temperature on the morphology and distribution of the precipitates of the further aged alloy, the representative TEM images of the aged alloys are presented in figure 6 to the figure 8. The aging for all as-homogenized alloys was conducted at 200 °C for 18 h. The figure 6 shows the TEM image of precipitates and corresponding selected area electron diffraction (SAED) patterns of the aged alloy under homogenization treatment at 500 °C for 8 h. It exhibited explicitly that the plate-shaped precipitates uniformly distributed in the α-Mg matrix. The length of plate-shaped precipitates was about 20 nm. Moreover, the distribution of precipitate was relatively sparse. As mentioned previously, lots of residual β phase existed in
grain boundary after the homogenization treatment at 500 °C for 8 h. The RE element were mainly concentrated on residual β phase at grain boundaries. Consequently, the concentration of RE elements in the matrix was not high enough, and only a limited number of RE atoms participated in the nucleation and growth of the precipitates.

The figure 7 provides the TEM image of precipitates and corresponding SAED patterns of the aged alloy under homogenization treatment at 515 °C for 8 h. As shown in figure 7(a), the plate-shaped precipitates were parallel to (1 1 0 0)α. The length of plate-shaped precipitates was about 50 nm. In order to further confirm type of the prismatic plate-shaped precipitates, the high magnification TEM image and corresponding SAED patterns were given in the figure 7(b). The incident electron beam direction was parallel to the [2 2 3 4]α. According to the SAED patterns, the prismatic plate-shaped precipitates could be identified as the β′ phase, which was the main strengthening phase in the peak aged Mg-RE alloys [20, 21]. The orientation relationship between β′ phase and α-Mg matrix was (1 1 0)β′ // (1 1 0)α, and [1 1 2]β′ // [2 2 4 3]α. Compared with the β′ phase in the aged alloy under homogenization treatment at 500 °C for 8 h, the length and number of β′ phase increased significantly. With the homogenization temperature raised from 500 °C to 515 °C, the eutectic β phase dissolved more adequately. In this regard, the concentration of RE elements in α-Mg matrix could increase conspicuously. It can effectively promote the precipitation of β′ phase. In addition, the alloy quenching from a relatively high temperature could induce more quenched-in vacancies, which can act as the effective nucleation site of precipitates during the early aging state by reducing the nucleation barrier [13, 22]. Meanwhile, the quenched-in vacancies could also accelerate the diffusion of solute atoms. According to the above observation and analysis, the increase of homogenization temperature from 500 °C to 515 °C was conducive to the nucleation and growth of the β′ phase during the aging.

The figure 8 shows the TEM images and corresponding SAED patterns of the aged alloy under homogenization treatment at 525 °C for 8 h. Some coarse β1 phase (marked by the red arrow) were observed in α-Mg matrix [23] (see the figure 8(a)). The β1 phase was larger than 100 nm in length and 30 nm in thickness. The plate-shaped β phase was parallel to the (1 1 0 0), and usually formed in the peak aged and the over aged Mg-Nd series and Mg-Sm series alloys [23, 24]. Observations with figure 8(b) revealed that the β′ phase is completely coherent with the α-Mg matrix. The previous OM observation suggested that the content of RE elements in α-Mg matrix remains at the same levels when the homogenization temperature further increased to 525 °C. However, with the increase of homogenization temperature, the concentration of quenched-in vacancies...
further increased. Hence, the precipitation process was accelerated, and the plate-shaped $\beta_1$ occurred in $\alpha$-Mg matrix.

3.3. Effect of homogenization temperature on mechanical properties

The relationship between the mechanical properties of the aged alloy and homogenization temperature are shown in the figure 9. The ultimate tensile strength (UTS) and yield strength (YS) of the aged alloy under homogenization treatment at 500 °C for 8 h were 232 MPa and 143 MPa. The homogenization temperature raised to 515 °C, both the values of UTS and YS reached to 261 MPa and 154 MPa, respectively. Accordingly, the elongation increased from 5.1% to 5.8%. Obviously, the precipitation strengthening is the most effective method to improve the performance of Mg-RE alloys. The homogenization temperature raised from 500 °C to 515 °C, the number of $\beta'$ phase increased significantly. The mechanical properties of the aged alloy were enhanced by subsequent aging treatment. In addition, although the grain size is smaller when homogenization treatment was insufficient, there were still some residual $\beta$ phase particles distribution along grain boundary. The residual $\beta$ phase particles easily induced stress concentration and micro-cracks. Hence, with the $\beta$ phase gradually dissolved into the $\alpha$-Mg matrix alloy, the tensile strength was increased. Meanwhile, the reduction of $\beta$ phase was conducive to the improvement of the elongation. However, when the homogenization temperature further increased, the tensile strength of aged alloy decreased obviously. Especially, the aged alloy after homogenization treatment at 525 °C for 8 h, the UTS was 233 MPa, which decreased by 10.7%; the YS was 145 MPa, which decreased by 5.8%, compared with the peak value. The elongation was reduced from 5.8% to 5.4%. According to the Hall-Petch relationship [25], when the homogenization temperature reached 525 °C, the coarse grains could deteriorate the mechanical properties of the aged alloy. Furthermore, the research results of Nie et al [26] revealed the relationship between morphology of precipitates and dispersion strengthening. The contribution of $\beta'$ phase on inhibiting the dislocation motion was stronger than that of $\beta_1$ phase. The similar phenomenon had been observed in the Mg-Nd-Zn-Zr and Mg-Gd-Zn alloys [27, 28]. For the peak-aged Mg-Nd...
and Mg-Gd series alloy, the major strengthening phase was the $\beta'$ phase. With the increasing the aging time, the metastable $\beta'$ phase transformed to $\beta_1$ gradually. The mechanical properties of the over-aged alloy were deteriorated. In this work, the tensile strength of aged alloy under homogenization treatment at 525 °C for 8 h decreased obviously, compared with peak value of the alloy. Moreover, the decrease in elongation could be attribute to coarse grain.

3.4. Fracture surface morphology
The figure 10 shows the typical fracture microstructure of the aged alloy. In order to better observe the change of $\beta$ phase content at the fracture surface. Two modes of fracture images were taken. The figure 10(a) to figure 10(c) and figure 10(g) to figure 10(i) are the secondary electron images, the figure 10(d) to figure 10(f) and figure 10(j) to figure 10(l) are the back scattering electron images. A large number of cleavage planes were observed in the fracture surface of aged alloy. The fracture morphology of the aged alloys was typical transgranular cleavage fracture. In addition, the size of the cleavage plane increased gradually with the increasing of homogenization temperature. The increasing of the size of cleavage plane was mainly related to the grain coarsening. Moreover, the content of $\beta$ phase of fracture surfaces decreased significantly with the homogenization temperature increased from 500 °C to 515 °C.

4. Conclusions
1. The as-cast alloy was mainly composed of $\alpha$-Mg matrix, $\beta$-Mg$_{12}$(Nd,Sm,Zn) phase and Zr-containing particles.
2. Upon increasing the homogenization temperature from 500 °C to 525 °C for 8 h, the average grain size of as-homogenized alloy increased from 76 μm to 156 μm. And the content of the β phase decreased gradually. The increase of homogenization temperature could accelerate the precipitation process.

3. The optimal homogenization temperature for the aged alloy was 515 °C, the YS and UTS of the aged alloy were 154 MPa and 261 MPa, and the elongation rate was 5.8%, respectively. The fracture surfaces of the aged alloy mainly exhibited a transgranular cleavage fracture.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

Author contributions

S Z, L W and E G conceived and designed the experiments; S Z and T M performed the experiments; T M, S Z Y C and Z W analyzed the data; S Z D L and J L acquired the funding and did the project administration; S Z, T M and C W prepared the original draft; S Z, T M and J L revised the paper and created the final version.

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Conflicts of interest

The authors declare no conflict of interest.

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