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ARTICLE

Effect of Homogenization on Microstructure and Mechanical Property of Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y Alloy

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Abstract: The microstructure evolution of Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy during homogenization was investigated by differential thermal analysis (DSC), optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction. Meanwhile, the Viker's hardness (HV) was tested. Results show that the microstructure of as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy consists of α-Mg, $Mg_3(Y,Nd)_2Zn_3$ (W), Zn-Zr, $Mg_{12}(Y,Nd)Zn$ (X) and $Mg_3(Y,Nd)Zn_6$ (I) phases. The as-cast sample has an endothermic peak at 510 ºC, which disappears in the alloy homogenized at 500 °C for 16 h. A small quantity of W phases dissolve at 470 and 490 ºC. However, after homogenization at 500 ºC for 16 h, there is only a small number of W, I and X phases, and dendritic segregation is almost eliminated. Therefore, the optimum homogenization parameter is 500 ºC for 16 h. Homogenization can effectively decrease the hardness of the as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy from 1852 MPa to 1442 MPa, so it is beneficial to further deformation of the alloy.

Key words: magnesium alloy; homogenization; microstructure; hardness

Lightweight magnesium (Mg) alloys have received considerable attention due to their fuel efficiency and green environment[1, 2]. In order to optimize microstructure and mechanical properties, rare earth (RE) elements such as yttrium (Y), lanthanum (La), cerium (Ce) and neodymium (Nd) were added to the magnesium alloys[2]. The report[3] reveals that element Y can refine the grain size in the as-cast Mg-Zn-Zr alloys, and improve the mechanical properties. Wu et al.[4] find that the strengthening effect of mixed Nd and Y is better than that of single Nd addition at room temperature and elevated temperature. However, during solidification, rare earth elements may result in severe compositional segregation and non-uniform microstructure which reduces the thermoplasticity and workability[5]. Homogenization can effectively reduce micro-segregation, stress concentration and the harmful effects of the eutectic structure, and improve workability[6-9]. L. Bao et al.[10] find that homogenization at 300 ^{o}C for 24 h can improve the microstructure of Mg-Li-RE-Zr alloy, including grain size, cellular dendrite and low melting-point particles. It is reported[11] that the mechanical properties of the Mg-15Gd-5Y-0.5Zr alloy were improved by heat treatment at 525 ^{o}C for 12 h.

It is well accepted that homogenization can effectively improve the microstructure and mechanical properties of the magnesium alloy. Unfortunately, the understanding of the as-cast and homogenized microstructures of Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y is lacking. Hence, in order to obtain the microstructure evolution during homogenization, Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy were studied under various heat-treatments.

1 Experiment

The materials in this study were prepared in the laboratory. Mg (99.93%), Zn (99.95%) and Mg-30Zr (wt%) were used in the experiment. Rare earth (RE) elements in the form of intermediate alloys of Mg-30wt%Nd and Mg-30 wt%Y were added to the metal at 750 ^{o}C under the

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protection of SF_6 and CO_2 gas mixture. Molten metal was poured into an iron mold at 700 °C. The ingot dimension is 60 mm×120 mm×300 mm. The chemical composition of the alloy is shown in Table 1.

The ingot was tested by a differential thermal analysis (DSC, STA 409 PC Luxx) with a heating and cooling rate of 10 ºC/min and the highest heating temperature was 605 ºC. In order to obtain the microstructure evolution during homogenization, the samples were homogenized at 470, 490 and 500 ºC for 4, 8, 12 and 16 h. In addition, another sample was treated at 500 °C for 20 h. Samples were heated in an electric resistance furnace with CO_2 gas protection. The heated samples were cooled in air.

Samples for microstructure observation were prepared by mechanical polishing and then etched in a solution of 4% nitric acid in ethanol for 5~10 s. The microstructures of the investigated alloys were observed by an optical microscope (OM, Axio Imager) and scanning electron microscopy (SEM, Hitachi LTD.S-3400N) equipped with an energy dispersive X-ray spectrometer (EDS). X-ray diffraction (XRD, D/MAX-2500/PC) was used for phase identification. The hardness was measured through a Vicker's hardness tester (HVS- 30Z/LCD). Load time was 15 s and 6 points were randomly chosen on each sample. The average values of hardness were obtained and the variances were calculated.

2 Results and Discussion

2.1 Microstructure of as-cast alloys

Fig.1 shows the microstructure of the as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy. The microstructure is characterized with an α-Mg matrix and intermetallic compounds which are continuously distributed along the grain boundary. In addition, a small number of W phases possesses a face-centered cubic with a = 0.6832 nm and a chemical formula of $Mg_3Y_2Zn_3$[12]. The atom radius of Y is 0.227 nm and that of Nd is 0.264 nm. The latter is larger than the former. Nd atoms dissolve into globular W phase, distort the lattice and unevenly disperse in the grain interior. Therefore, the crystal structure of $Mg_3(Y,Nd)_2Zn_3$ phase is the same as that of W-phase and the lattice parameter is a = 0.695 nm which is slightly bigger than that of W-phase[13]. The XRD pattern of the as-cast sample is shown in Fig.2a. The XRD pattern of the as-cast sample indicates that the phases mainly consist of α-Mg, Zn-Zr, W phases and a small amount of I and X phases. Fig.3 shows the SEM micrograph and the distribution of the main elements Zn, Zr, Y, and Nd in as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy. It can be seen that these elements are mainly enriched in the grain boundary, and partial Zr is distributed in the grain interior. The intermetallic compound in grain boundaries is $Mg_3(Y,Nd)_2Zn_3$ (W) phase and it causes much dendritic segregation in grain boundary. What is more, it can be seen that globular phase in grain (in Fig.4a) contains the element of Mg, Zn and Zr from Fig.4b, and the ratio of Zr to Zn is close to 1:1. The globular phases in grain interior are Zn-Zr.

Table 1 Chemical composition of Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy (wt %)

Mg	Zn	Zr	Y	Nd	Others
89.507	5.867	1.623	0.858	1.569	0.576

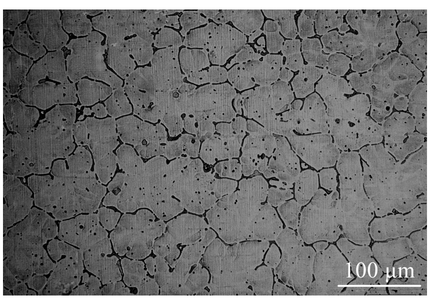


Fig.1 Microstructure of as-cast alloy

2.2 DSC results of the as-cast and homogenized alloy

The DSC curve (Fig.5a) of the as-cast alloy reveals that the endothermic peak occurs at 510 ºC and the eutectic phase dissolves at 502.3 ºC. High temperature may cause microstructure to abnormally grow-up or over-heat during homogenization. Thus, the highest temperature for homogenization should be lower than the melting point (502.3 ºC) of eutectic phase.

Fig.5b shows the DSC curve of the sample homogenized at 500 ºC for 16 h. Endothermic peak disappears in a temperature range from 502.3 ºC to 517 ºC. The XRD pattern of the sample homogenized at 500 ºC for 16 h is shown in Fig.2b. The peak can be indexed as α-Mg and a small amount of I and X phases, which prove that the main eutectic phase almost dissolves in the matrix.

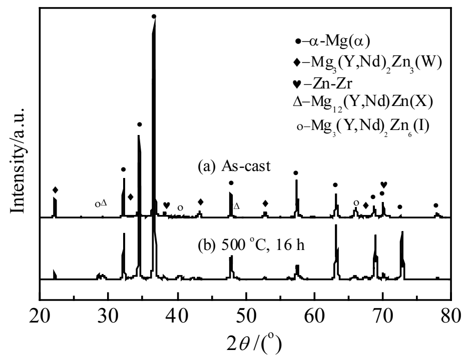


Fig.2 XRD patterns of as-cast alloy (a) and as-homogenized alloy at 500 ºC for 16 h (b)

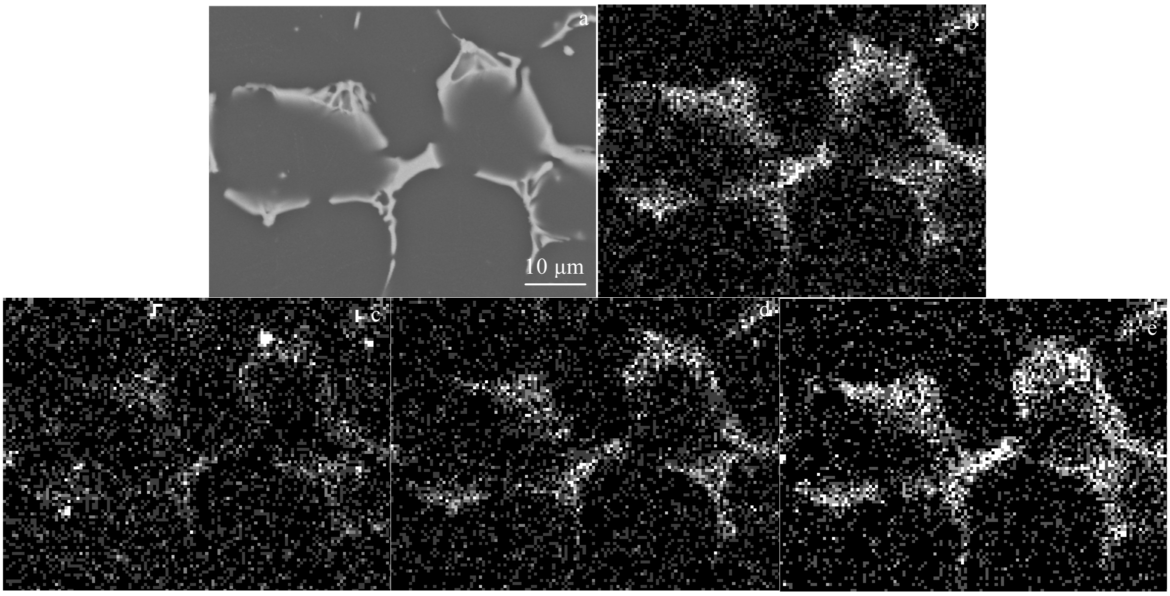
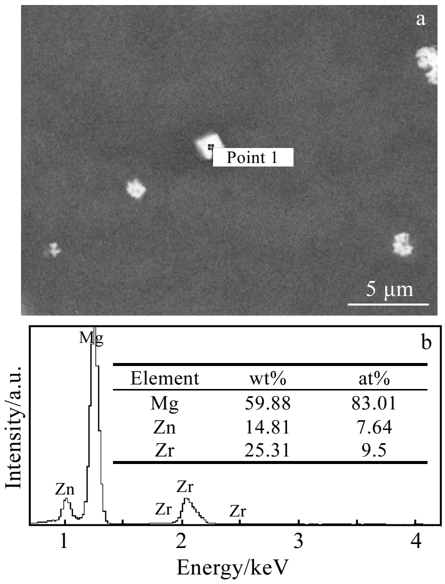


Fig.3 Microstructure (a) and elements distribution (b~e) of as-cast alloy: (b) Zn, (c) Zr, (d) Y, and (e) Nd



Element	wt%	at%
Mg	59.88	83.01
Zn	14.81	7.64
Zr	25.31	9.5

Fig.4 SEM image (a) and EDS analysis of point 1(b)

2.3 Microstructure evolution during homogenization

Fig.6 shows the microstructure of the Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy homogenized under different conditions. Fig.6a, 6b and 6e present the microstructure of the alloys homogenized at different temperatures for 16 h. From the microstructure of the sample homogenized at 470 ºC for 16 h (Fig.6a), it can be noted that a small amount of second phases have dissolved, compared with that of the as-cast alloy (Fig.1). With the homogenization temperature increasing, the continuous networks of eutectic compounds begin to become discontinuous (Fig.6b). When the temperature is 500 ºC (Fig. 6c), micro-segregation is almost eliminated, and there only remain a few small particles with high melting point in the grain boundary.

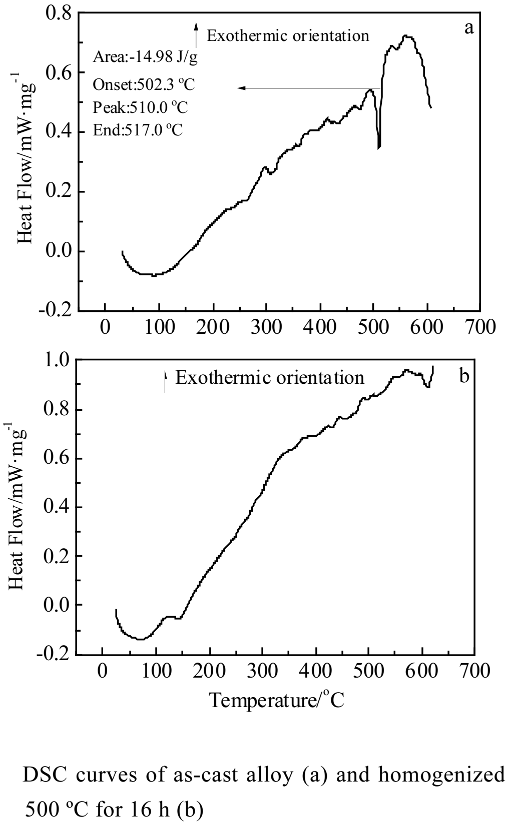


Fig.5 DSC curves of as-cast alloy (a) and homogenized alloy at 500 ºC for 16 h (b)

Fig.6c, 6d and 6e show the microstructures of the alloy homogenized at 500 ºC for various holding-time. The

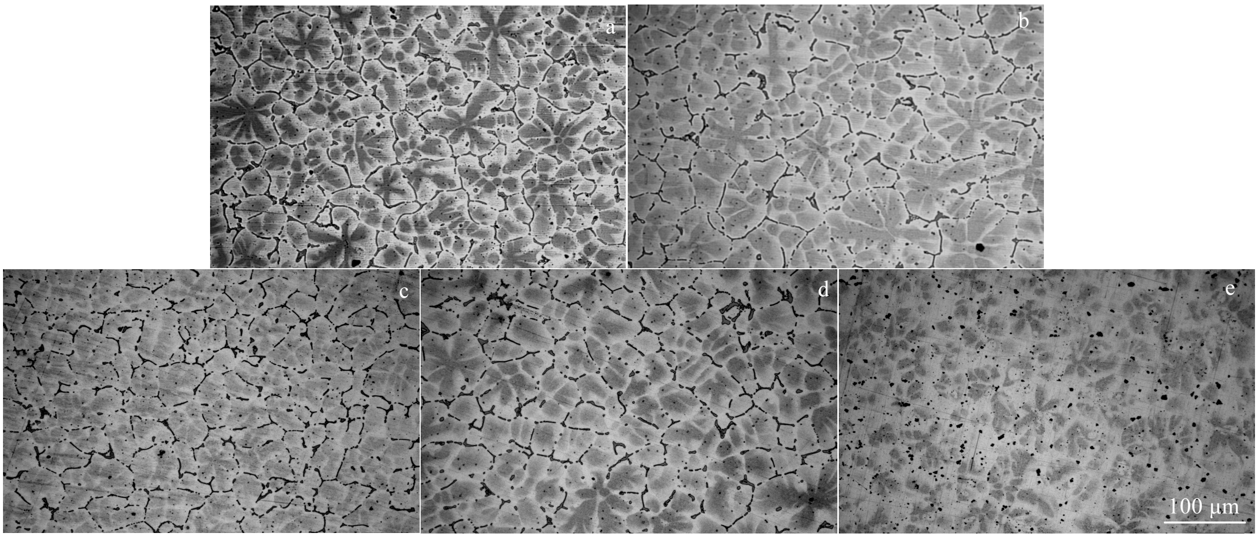


Fig.6 Microstructures of alloy homogenized under different conditions: (a) 470 ℃, 16 h; (b) 490 ℃, 16 h; (c) 500, 8 h; (d) 500, 12 h; (e) 500, 16 h

microstructure of the alloy homogenized at 500 ºC for 8 h (Fig.6c) is similar to that in as-cast alloy. With the holding-time prolonging, the second phases become discontinuous (Fig.6d). The dissolution of the eutectic structure is significant at the temperature of 500 ºC for 16 h (Fig.6e), less second phases remain on the grain boundary, and microstructure becomes uniform.

According to the first law of diffusion, the relationship between the diffusion coefficient and temperature can be summarized as Arrhenius formula[14]. With the increase of temperature, the diffusion coefficient increases, and atomic thermal activation is more apparent. Due to higher diffusion rate, the time to achieve the homogenization is shorter. Thus, the homogenization temperature should be increased as much as possible.

Microstructure of the sample homogenized at 500 ºC for 16 h was further investigated by SEM and EDS. Fig.7 presents the surface scan of the sample homogenized at 500 ºC for 16 h. Comparing Fig.7 with Fig.3, Zn and Zr elements are more uniformly distributed in the matrix, and few Y and Nd elements exist in the grain boundary. The EDS results shown in Fig.8 and Table 2 demonstrate that the particles mainly consist of Y and Nd elements. Combining EDS analysis (Table 2) with XRD result (Fig.2b), we can conclude that the spherical particles are $Mg_3(Y,Nd)_2Zn_3$ phase. Such RE-rich particle were found in other homogenized Mg-RE alloy[15-17]. It is found that the RE-rich particles distributed randomly at the grain boundaries or in the grain interior mainly form during high temperature heat treatment[16]. Zhang et al.[18] believe that the solid solubility of RE elements in α-Mg matrix increases remarkably with the temperature increasing. The dissolution of the second phases at the grain boundaries and the diffusion of RE atoms cause the increase of RE concentration in α-Mg matrix when the alloy is homogenized at high temperature for a long time.

2.4 Effect of homogenization treatment on hardness

Microstructure and mechanical properties are changed during homogenization treatment. To some extent, hardness values can confirm the dissolution of constituent particles. In addition, the hardness value is generally used to choose the homogenization process. Therefore, the Vicker's hardness values of samples homogenized under various condition were tested in this study. For each sample, the average hardness value (HV_1) is obtained by randomly selecting 6 points and the variance (σ^2) is calculated. These hardness values are presented in Table 3.

These second phases play an important role in pinning dislocation when the samples deform during hardness test. The hardness value of samples with a large number of second phase becomes higher. A large number of rare earth elements (Y, Nd) are enriched in grain boundary and form segregation during solidification. The segregation in as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy is so serious that hardness value reaches 1852 MPa and variance is 114.1. At the beginning of the homogenization treatment, the concentration gradient of alloy elements and the atomic diffusion rate are large, and the eutectic phases on the grain boundary promptly dissolve; thus the hardness value rapidly decreases. As the homogenization time extends, the decomposition of the eutectic phase decreases the hardness and the alloy element dissolving into matrix increases the

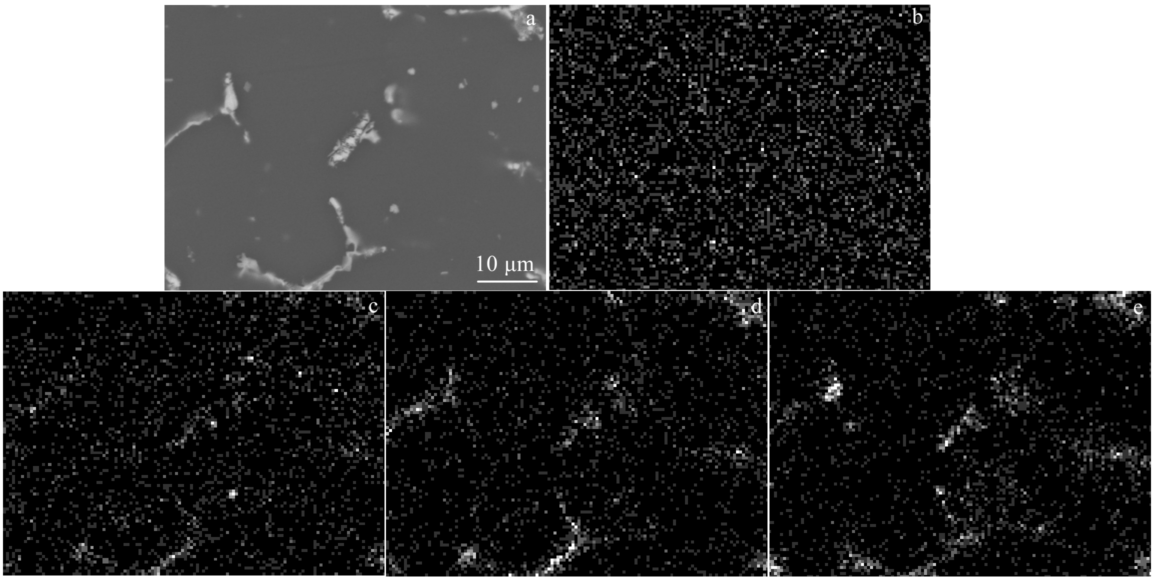


Fig.7 SEM image (a) and elements distribution (b~e) of the sample homogenized at 500 °C for 16 h: (b) Zn, (c) Zr, (d) Y, and (e) Nd

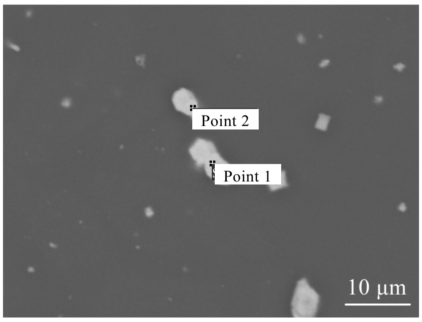


Fig.8 EDS analysis positions of homogenized alloy after 500 °C for 16 h

Table 2 EDS analysis of homogenized alloy after 500 °C for 16 h of point 1 and 2 in Fig.8

Element	Mg	Zn	Y	Zr	Nd
Point 1	2.30	2.05	11.05	4.87	79.73
Point 2	2.89	2.33	9.88	0	84.90

hardness, which causes little decrease of hardness value. For the alloy homogenized at 500 °C for 16 h, hardness value and variance drop to 1442 MPa and 11.4, respectively. It is noted that homogenization significantly affects the hardness, and enhance the ability of plastic deformation.

Table 3 Result of Vickers hardness (HV_1) and variance (σ^2) of samples at different condition

Cond	As-cast	470 °C				490 °C				500 °C			
		4 h	8 h	12 h	16 h	4 h	8 h	12 h	16 h	4 h	8 h	12 h	16 h
HV_1/MPa	1852	1569	1539	1522	1512	1545	1513	1487	1471	1533	1494	1467	1442
σ^2	114.1	71.9	47.3	36.8	30.8	38.0	32.2	28.4	23.7	26.8	26.1	23.1	11.4

3 Conclusions

1) For as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy, main dendrite intermetallic compounds are $Mg_3(Y,Nd)_2Zn_3$ phase and globular phases unevenly distributed in the matrix are Zn-Zr phase. In addition, there are a small amount of I and X phase.

2) The non-equilibrium phases gradually dissolve with increasing homogenization temperature or time. After homogenization treatment at 500 °C for 16 h, there are merely a few $Mg_3(Y,Nd)_2Zn_3$ dispersing at grain boundary, and dendritic segregation is almost eliminated. 500 °C for 16 h is considered as the best homogenization parameters.

3) Homogenization treatment can effectively reduce the hardness of the as-cast Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y alloy from 1852 to 1442 MPa, and enhance the ability of plastic deformation of material.

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均匀化处理对 Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y 合金组织和硬度的影响

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摘 要：采用差热分析(DSC), 金相显微镜(OM), 扫描电镜(SEM), 能谱分析(EDS)和 X 射线衍射研究了 Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y 合金均匀化过程中微观组织演化；另外，测试了维氏硬度。研究发现，铸态 Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y 合金包含：α-Mg, $Mg_3(Y,Nd)_2Zn_3$ (W), Zn-Zr, $Mg_{12}(Y, Nd)Zn$ (X)和 $Mg_3(Y, Nd)Zn_6$ (I)相。铸态试样的吸热峰在 510 ℃，合金经 500 °C, 16 h 均匀化处理后吸热峰消失。经 470 和 490 °C 均匀化处理后，少量的 W 相溶解；而经过 500 °C, 16 h 均匀化处理后，仅有少量 W, I 和 X 相存在，枝晶偏析基本消除，最佳均匀处理工艺为 500 °C, 16 h。均匀化处理可以将 Mg-5.9Zn-1.6Zr-1.6Nd-0.9Y 合金的硬度从 1852 降到 1442 MPa，这样有利于后续塑性变形。

关键词：镁合金；均匀化；微观结构；硬度

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