Analysis on micro-structure and mechanical properties of Mg-Gd-Y-Nd-Zr alloy under as-cast or aging state and its reinforcement mechanism

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Abstract: Gd in Mg-RE liquid alloy has a high solid solubility, which can rapidly decreases with the drop of temperature. Thus the solid solution and aging reinforcement of liquid Mg alloy can be achieved. The micro-structure and mechanical properties of Mg-Gd-Y-Nd-Zr alloy under as-cast and aging state were studies by composition analysis, micro-structure observation and mechanical properties test, and the reinforcement mechanism of mechanical properties was analyzed. Results show that the micro-structure of as-cast alloy is mainly composed of α -Mg matrix and eutectic phase. The dendritic eutectic phase exhibits a discontinuous mesh distribution along the grain boundary. And the average grain size of alloy is about 46 μ m. After aging treatment, the average grain size is 52 μ m and the eutectic phase can dissolve into α -Mg matrix completely. The massive particle phase distribute in grain boundary and their number increased significantly, which can strengthen the alloy effectively. The tensile strength and yield strength of alloy are improved significantly from as-cast to aging state. But the elongation shows a decreasing trend. The Gd after dissolved into α -Mg matrix can lead to serious lattice distortion, improve the antiphase domain boundary and further improve the alloy yield strength.

Key words: Mg-Gd-Y-Nd-Zr alloy; micro-structure; mechanical properties; strengthening mechanism

0 Introduction

The micro-structure performance and mechanical properties of melt alloy can be improved by adding certain amount of rare earth elements in it, especially in the area of oxidation resistance and creep. So the application researches on rare earth elements in Mg-RE alloy attract scholars' attention [1-5]. The rare earth element Gd in M-RE liquid alloy has a high solid solubility and can rapidly be weakened with the decreasing of temperature, which can realize a time limitation of melt Mg alloy, so Gd is considered to a typical rare earth element to improve the performance of Mg alloy [6-7]. Marco Fronzi et al studied the mechanical properties of Gd in M-RE and found that the Mg-Gd formed by Gd and M-RE has a high hardness and melting point, which can effectively improve the mechanical strength and creep resistance [8]. F Mokdad et al studied the mechanical properties of Zr in M-RE and found that it can effectively hinder the dislocation movement, and the grain growth also enhanced the mechanical properties of Mg alloys [9].

Because of the aging strengthening for Mg-Gd series alloy, researchers improve the mechanical properties of Mg alloy by using aging treatment ways [10]. A number of studies show that the primary β'' (Mg₃Gd) phase and β' (Mg₇Gd) phase coexists in the Mg substrate in Mg-Gd series alloy with a 120° angle spindle uniform distribution [11]. The alloy can have a very strong stability in a larger temperature range, which strengthens the alloy significantly and improves the performance of creep. Fine-grain strengthening mechanism has been widely applied in Mg-Gd alloy, and there are two ways to achieve the purpose of fine grains usually: (1) Adding grain refiner such as Nd and Zr to Mg-Gd alloy in the preparation process can increase the alloy intensity. Mg₁₂Nd phase with high hardness and melting point precipitated by adding Nd can effectively improve the strength and creep properties of alloy; Zr can refine as-cast organization significantly. (2) Adopting Some deformation process such as plastic deformation, thermal deformation process: the nano-scale grain of Mg-Gd alloy can be obtained after ECAP processing with α -Mg grain 0.6~1.0 μ m, even it may be realized the superplasticity at low temperature [12-13].

On the basis of related literature researches[14-20], the micro-structure and mechanical properties of

Mg-Gd-Y-Nd-Zr alloy under as-cast and aging state were studied via composition analysis, micro-structure observation and mechanical properties test, and the reinforcement mechanisms of mechanical properties were analyzed.

1 Experimental materials and methods

These dried materials include pure magnesium ingot (99.95%) and master alloy Mg-30Gd, Mg-10Y, Mg-25Nd, Mg-5Zr, and the chemical composition of the tested alloy is shown in [table 1](#). The melted alloy in induction furnace melting was casted under environment of 1vol.%SF₆+99vol.%CO₂. And the alloy was heated continually until 780°C. Keeping 780°C for 10 min, and then pouring the solution alloy into the mould with preheated 250°C. The alloy ingot casting was obtained after pouring shape and the protective coating avoiding of oxidation is prepared by using MgO powder. Then the aging (225°C×16h) of related processing is done through the box-type furnace.

The micro-structure and mechanical properties of Mg-Gd-Y-Nd-Zr alloy were observed and tested by using the Olympus optical microscope, JSM-5610LV scanning electron microscope (SEM), JEM-2100 high resolution electron microscopy (HRTEM). The grain size of tensile fracture morphology for alloy sample under room temperature were measured through the cutting line method, the micro composition were analyzed by using the scanning electron microscopy with energy spectrometer (EDS); and the phase analysis were done through the X-ray diffractometer (XRD) with D8Advance type. The relevant tensile test were made in the island ferry AG-I250kN precision universal experimental machine with the tensile speed 1mm/min.

Table 1 Chemical composition of tested alloys (mass fraction, %)

Alloy	Gd	Y	Nd	Zr	Mg
Mg-10Gd-Y-2.5Nd-0.5Zr	10.25	1.06	2.78	0.55	Bal.

2 Results and analysis of micro-structure

2.1 Micro-structure

In order to grasp the grain distribution and grain size more accurately, [Fig. 1](#) shows the micro-structure of Mg-Gd-Y-Nd-Zr alloy under as-cast or aging state. The micro-structure of as-cast alloy is mainly composed of α -Mg matrix and eutectic phase, the dendritic eutectic phase has a discontinuous mesh distribution along the grain boundary. And the average grain size of alloy is about 46 μ m. Due to the sharp temperature drop, the new produced primary dendritic could not grow up under the eutectic reaction temperature. The liquid phase among these primary dendritics began to react and formed the eutectic phase. At this time, a discontinuous reticular distribution formed as the increasing of eutectic phase.

[Fig. 1\(b\)](#) shows that the micro-structure of Mg-Gd-Y-Nd-Zr alloy with aging state, which is mainly composed of α -Mg matrix and undissolved phase with irregular shape (block or strip). The mesh dendritic compound disappeared by compared with that of as-cast state alloy, and a possible reason is that such irregular shapes are unstable under 525°C. The twin crystal partly appears in the organization of aging state alloy. There are the lump and granular phase distribution in the grain boundary or intracrystalline. The average grain size of alloy is 52 μ m after the aging treatment.

Fig.1 Micro-structure of alloy: (a)as-cast and (b) aging state

Fig. 2 shows the XRD test results of Mg-Gd-Y-Nd-Zr alloy. The as-cast organization includes α -Mg substrate, Mg_5Gd phase and $Mg_{12}Nd$ phase. The eutectic phase is supposed to be regarded as the product of quasi-binary eutectic reaction ($L-Mg \rightarrow \alpha-Mg + \beta-(Mg_5Gd + Mg_{12}Nd)$). This indicates that the XRD diffraction peak under as-cast comes from the α -Mg matrix and $\beta-(Mg_5Gd + Mg_{12}Nd)$ phase. In the aging process, the eutectic phase gradually dissolved into Mg substrate. When the aging treatment is finish, the eutectic phase dissolved into Mg substrate completely. Thus, the XRD diffraction peak of residual eutectic phase is not obvious.

Fig. 2 XRD of alloys: (a)as-cast and (b) aging state

The SEM morphology of alloys under as-cast or aging state can be seen in Fig. 3. The bulky as-cast dendrite distributes along the grain boundary; a few granular dissolution phases in aging state are distributed in the grain boundary or intracrystalline; the aging precipitate particles increased obviously and its distribution is along the grain boundary. After aging treatment, the Mg_5Gd phase precipitate is near the grain boundary and its granulars form into a square. The precipitation method of $Mg_{12}Nd$ phase is unchanged. This indicates that the β phase (square phase) is changed sharply from the dendritic into granulate in the process of aging treatment, as a result of the desolventizing way change of supersaturated Gd atomic and the increasing diffusion rate of Gd atomic.

Fig.3 SEM micro-structure image of alloys: (a)as-cast and (b) aging state

In Fig. 3(b), it is clear that some black cube particles go through SEM in the grain boundary or intracrystalline. These black particles present a shape of square phase by observing the SEM image. Selecting a single square along to be analyzed by EDX, as shown in Fig. 4. The square phase is a compound with rich Gd and small amount of Gd, Nd, Zr. After aging treatment, the eutectic phase almost dissolved into all α -Mg matrix, but the number of square phase increased significantly. There may be two reasons to explain the occurrence of these squares:

- (1) A few square phases had formed in casting process and did not dissolve in α -Mg matrix due to the thermal stability, meanwhile the eutectic compounds of rare earth element spreaded to the square phase and made it grow.
- (2) Due to the recombination between rare earth element Gd, Nd and metal element Mg, Zr, the new square

phase is formed, which increases the amount of squares phase.

The assemble of squares phases can cause stress concentration and crack germination, which affect the strength and plasticity. There is no exact conclusion about the effect of square phase on mechanical properties of alloy currently.

Fig.4 SEM image and EDX spectrum of cuboidal particles: (a) SEM image and (b)EDX spectrum

3 Mechanical properties and strengthening mechanism

3.1 Result analysis of mechanical properties

In general, the mechanical properties of Mg alloy have a close relationship with the strengthening phase distribution, the uniformity of internal organization and grain size. The intermetallic compound Mg_5Gd (eutectic temperature 542 °C) generated from the interaction between Gd and Mg has the advantages of high hardness and sound thermal stability, which can improve the mechanical properties of alloy at room temperature or high temperature.

Alloys have different mechanic properties with different temperatures. Table 2 lists the tensile mechanical properties of Mg-Gd-Y-Nd-Zr under as-cast and aging state at room temperature 20°C and high temperature 300°C. The measured three main parameters are tensile strength σ_b , yield strength $\sigma_{0.2}$ and elongation δ . At room temperature, the tensile strength and yield strength of alloy are improved significantly from as-cast to aging state with σ_b changing from 115.7MPa to 181.8MPa and $\sigma_{0.2}$ from 69.6MPa to 124.1MPa. But the elongation shows a decreasing trend with δ from 10.47% to 8.07%. The changes of three parameters with high temperature 300 °C are more obviously by comparison with those of room temperature 20°C.

Table 2 Mechanical properties of alloys

temperature		20°C			300°C		
parameter	δ	σ_b	$\sigma_{0.2}$	δ	σ_b	$\sigma_{0.2}$	
	/%	/MPa	/MPa	/%	/MPa	/MPa	
as-cast	10.47	115.7	69.6	18.07	110.8	75.9	
aging state	8.07	181.8	124.	6.67	276.9	231.8	

The alloy after aging treatment will optimize its properties, such as uniform micro-structure, refined grain and higher dispersion degree of precipitated square phase, which can effectively prevent dislocating movement. Thus the mechanical properties of strengthening fine-grain and dispersion improved. Compared with the Mg matrix elements ($r_{Mg}=0.16nm$), the atomic radius of Gd ($r_{Gd}=0.18nm$) is larger. The contrast of atomic radius between them can obviously improve the strengthening effect of aging treatment. The solid solubility of Gd drops quickly with the decreasing of temperature, so the alloy is easy to form a supersaturated state. After aging treatment, the precipitation strengthening phase with evenly diffuse distribution be embedded some irregular dendritic hard particles on the soft Mg substrate.

During the tensing process, these irregular dendritic hard particles hinder the rotation of inter-granular and

further produce a new dislocation among the internal grains. The dislocation began to move with the increasing of load. But its movement will be hindered by the precipitated phase when the dislocation moves to precipitate phase interface. So a dislocation pileup happens and then causes stress concentration effect. When the stress concentration reaches the strength limit of precipitated phase, the precipitated phase will began to craze.

By the SEM morphology observation of tensile fracture at room temperature (Fig. 6), it is found that there are a large number of tear ridges and reconciliation steps appearing on the specimen fracture of as-cast alloy. A layer of boundary cracks and laminar morphology appear in the uneven fracture morphology. This is due to the large amount of thick dendrite shape layer and lamellar eutectic in the as-cast alloy, which can cause strong concentration leading to crack extension, a typical brittle fracture characteristics.

The tear ridges decreases and the reconciliation increases with the development of aging treatment. Because the dissolved dendrite and eutectic organization of alloy show a homogenization law and the stress concentration can be released. During the process of tensing, a plenty of tiny reconciliations are produced, which may cause the fracture morphology appear as a certain plastic deformation of cleavage fracture.

Fig.6 SEM morphology of tensile fracture at room temperature: (a)as-cast and (b)aging state

3.2 Strengthening mechanism analysis

3.2.1 Solid solution strengthening

In the Mg-Gd alloys, the Gd can form the unordered solid solution by using the method of substitution. The Gd in Mg matrix have a high solid solubility and big atomic radius. When it is squeezed into Mg matrix, massive particle phases with cube squares are produced. The central area with solute atoms can form stress concentration and affect the sliding surface roughness and hinder the dislocation movement. Thus the Mg-Gd alloys can improve the mechanical properties. The dislocation movement will be impeded when meeting the solute atoms under the action of external force after the solid solution strengthening. Assumed that the solute atoms are motionless at a lower temperature, the dislocation line is segmented by the solute atoms when it meets the solute atoms, which results from the solid solution strengthening.

After the Gd soluble into the Mg substrate, the supersaturated solid solution is formed. The atomic radius $R_{Gd}(0.1802nm) > R_{Mg}(0.159nm)$, is slightly lower than the limit conditions of forming substitutional solid solution (Hume Rothery rules ($\Delta R/R \leq 15\%$) [21]). So the Gd in Mg alloy has an obvious aging strengthening effect.

3.2.2 Coherent strengthening

Due to the difference lattice constant between β' precipitated phase and α -Mg matrix phase, β' precipitated phase in the aging state has a half coherent relationship with α -Mg matrix phase. So around the the lattice constant will produce a high elastic stress field, which can hinder the dislocation movement and produce the coherent strengthen. The larger the difference lattice constant is, the higher the lattice mismatch degrees $[(\alpha_{\beta'} - \alpha_{Mg}) / \alpha_{Mg}]$ is, the stronger the stress field around β' is, the more significant the strengthening effect of alloy is.

The β' precipitated phase with a good heat stability has a dense triangular distribution within the alloy matrix, which can effectively hinder the dislocating motion and increase the alloy strength. The influence of coherent strengthen mainly relates to antiphase boundary energy of β' precipitated phase, volume fraction, average size and lattice distribution [22-23]. Generally speaking, Mg alloy can improve the antiphase boundary energy by adding the Gd, thus the yield strength of alloy is improved.

4 Conclusion

1) The organization of Mg-Gd-Y-Nd-Zr alloy under as-cast state presents a bulky dendritic crystal and has a intermittent network distribution. After aging treatment, most branches dissolves into Mg substrate, only a small amount of particles and cubes are non-dissolved. And the average grain size is 52 μ m and the eutectic phase can completely dissolve into α -Mg matrix.

2) The tensile strength and yield strength of alloy are improved significantly from as-cast to aging state. But the elongation shows a decreasing trend. The Gd after dissolved into α -Mg matrix can lead to a serious lattice distortion and improve the antiphase domain boundary, which can improve the alloy yield strength.

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