Effect of Nd content on microstructure and mechanical properties of as-cast Mg-12Li-3Al alloy

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Abstract: In order to improve the mechanical properties of Mg-Li alloy with single β phase structure, Mg-12Li-3Al-xNd ($x=0.3, 0.7, 1.1, 2.0\text{wt.}\%$) alloy was prepared. Subsequently, the as-cast microstructure and mechanical properties were observed and tested. The results showed that the structure of Mg-12Li-3Al-xNd ($x=0.3, 0.7, 1.1, 2.0\text{wt.}\%$) as-cast alloy was composed of β phase matrix and Al_2Nd , $\text{Al}_{11}\text{Nd}_3$, MgLiAl_2 , Al_4Li_9 and AlLi phases. With the increase of Nd content in the alloy, the Al-Nd intermetallic compounds have a trend to change from needle-like $\text{Al}_{11}\text{Nd}_3$ to granular Al_2Nd . The hardness of as-cast Mg-12Li-3Al-xNd ($x=0.3, 0.7, 1.1, 2.0\text{wt.}\%$) alloy was stable at room temperature. The tensile strength of Mg-12Li-3Al-1.1Nd was as high as 180 MPa, the elongation rate of Mg-12Li-3Al-0.7Nd reached 53.7%, and the comprehensive mechanical properties of Mg-12Li-3Al-2.0Nd was the best. PLC phenomenon occurred during the tensile process of the alloys at room temperature. Therefore, the β -based Mg-Li alloy with good plasticity as well as enhanced strength can be obtained by a moderate addition of Nd and Al.

Key words: Mg-Li; β ; Nd; microstructure; mechanical properties

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As the lightest metal structural material today, magnesium lithium alloy is widely used in aerospace and military fields, and has broad application prospects in the fields of automobile, electronics, medical products and so on^[1]. Although magnesium lithium alloy has many excellent properties such as high strength and high stiffness ratio, strong cold and hot deformation ability and no obvious anisotropy, but it has also some obvious weaknesses. For example, the absolute strength is low, creep resistance and corrosion resistance are poor^[2,3], so strengthening the magnesium lithium alloy has become a key to expanding its application fields. Scholars at home and abroad have done much work on how to strengthen Mg-Li alloy^[4-6].

It can be seen from the binary-phase diagram of Mg-Li alloy that, according to the difference of Li content, Mg-Li alloy has three kinds of structure, i.e. α phase, $\alpha+\beta$ phase and β phase^[7]. When the lithium content is greater than 10.3%, the structure is single β phase (body-centred

cubic). The plasticity of β phase is much higher than that of α phase; its density is $1.3\text{--}1.5\text{ g}\cdot\text{cm}^{-3}$, and with the increase of Li content, its density linearly decreases. LA141A is a typical commercial magnesium lithium alloy with single β phase structure available today. But β is a softer phase compared with α , and creep of β -based Mg-Li alloy is likely to occur even when the stress is small, since its strength is low despite of the good plasticity.

Nd is one of the most prominent rare earth elements, and has good purification, modification and alloying effects on many nonferrous metal materials^[8]. The addition of Nd in magnesium or aluminum alloy can effectively improve air tightness, corrosion resistance and mechanical properties^[9-12]. Therefore, this study intends to prepare β -based Mg-Li alloy with good plasticity as well as enhanced strength by adding Nd and Al element. Furthermore, this study has some reference value for expanding the application of Mg-Li alloy.

1 Composition design and experimental procedures

1.1 Content of Li, Al and Nd

Although aluminum element has good solid solution

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strengthening effect on Mg-Li alloys, with the increase of Al content, the ductility of Mg-Li alloy decreases. Moreover, in a certain temperature range, when the mass fraction of Al is greater than 4%, the β single phase region will disappear^[13]. Therefore, in this study, using LA141A as a reference, the alloy prepared was expected to have not only good ductility due to single β phase structure, but also enhanced tensile strength, so the content of Li and Al were determined to be 12% and 3%, respectively.

Rare earth elements have low solid solubility in Mg-Li alloy, but can refine the structure of Mg-Li alloy and form a variety of intermetallic compound, which can increase the strength and improve the thermal stability of Mg-Li alloy^[14]. In addition, rare earth elements can narrow the solidification temperature range of the alloy and improve the compactness of the casting^[15, 16]. Nevertheless, the excess addition of rare earth will lead to the coarsening of grains and the significant increase of density for Mg-Li alloy, hence the content of Nd is determined to be 0.3%–2.0% in this study.

1.2 Materials

The main raw materials were pure magnesium ingot (Mg $\geq$ 99.95%), pure lithium of battery grade (in fragmented, Li content $\geq$ 99.9%), bulk Mg-25Nd master alloy and Φ 5 mm aluminum wire (Al $\geq$ 99.9%).

1.3 Methods and equipment

The alloy was melted in a vacuum resistance melting furnace (Model: ZRRL-M8). The mold should be prepared with coating

and preheated to about 300 °C before casting. The specific gravity was measured by the Archimedes method, and the quality of the alloy was measured by a balance with an accuracy of 1/10000 grams.

The microhardness of the alloy prepared was determined by a Digital Micro-hardness Tester. The test load was 980.7 mN (100 gf) and the holding time was 30 s. Each sample was randomly detected at 5 points and the results were averaged. The microstructure was observed and analyzed using SEM (scanning electron microscopy, PHILIPS XL30W/TMP, scanning voltage 20 kV) and EDS (energy dispersive spectrometer). The phase analysis of the alloy was conducted by means of X-ray diffraction apparatus (D/max-2500). The tensile properties at room temperature of the as-cast alloy were measured on a numerical controlled thermal physical simulation machine (Gleeble1500D). The test load was 8,160 kg and the tensile speed was 1.2 mm·min⁻¹.

2 Results and discussion

2.1 Composition and density

According to the addition amount of rare earth element Nd, four kinds of alloy were designed and prepared. The chemical composition and density of the alloys are shown in Table 1. As can be seen from Table 1, the actual composition of the Mg-Li-Al-Nd alloy prepared is basically close to that of the design. The density of alloys is relatively low on the whole, and the density of #1 alloy is the lowest.

Table 1: Chemical composition and density of alloy

Number of alloy	Alloy	Li (wt.%)		Al (wt.%)		Nd (wt.%)		Mg (wt.%)	ρ (g·cm ⁻³)
		Design content	Actual content	Design content	Actual content	Design content	Actual content		
#1	Mg-12Li-3Al-0.3Nd	12	13.02	3	3.44	0.4	0.30	bal.	1.4759
#2	Mg-12Li-3Al-0.7Nd	12	11.72	3	2.88	0.8	0.72	bal.	1.4936
#3	Mg-12Li-3Al-1.1Nd	12	13.21	3	3.10	1.2	1.05	bal.	1.5018
#4	Mg-12Li-3Al-2.0Nd	12	11.66	3	2.74	2.0	1.99	bal.	1.5351

2.2 Metallographic structure

Figure 1 shows the SEM micrograph of Mg-12Li-3Al-0.3Nd alloy. It can be seen from Fig. 1 that the matrix is single phase structure with some gray particles on it. XRD analysis results in Fig. 2 further indicate that the matrix of the alloy is single phase β (Li), the gray particles in Fig. 1 are MgLiAl₂, AlLi and Al₄Li₉, which give a precipitation strengthening effect on the matrix. In addition, there is a small amount of white acicular phase in the matrix, which is Al₁₁Nd₃ and has been confirmed by relevant literature^[10,12].

Figure 3 shows the SEM micrograph of Mg-12Li-3Al-0.7Nd

alloy and the EDS analysis of the white granular phase. It can be seen that there is much Al and Nd in the white granular phase. Due to the small particle size and large amount of Mg in the matrix, the peak of Mg was obtained in the EDS analysis. Figure 4 shows the XRD results of Mg-12Li-3Al-0.7Nd alloy, which indicates that the matrix is composed of single phase β (Li), containing Al₂Nd, Al₁₁Nd₃ intermetallic compounds and a small amount of MgLiAl₂ and Al₄Li₉. Combined with the EDS analysis results, it can be inferred that the white granular phase in Fig. 4(a) is Al₂Nd, the small amount of white acicular phase was Al₁₁Nd₃ and the gray particles were MgLiAl₂ and Al₄Li₉.

Fig. 1: Microstructure of Mg-12Li-3Al-0.3Nd alloy

Fig. 2: X-ray diffraction pattern of Mg-12Li-3Al-0.3Nd alloy

Fig. 3: Microstructure (a) and EDS analysis (b) of Mg-12Li-3Al-0.7Nd alloy

Fig. 4: X-ray diffraction pattern of Mg-12Li-3Al-0.7Nd alloy

Figure 5 shows the SEM micrograph of Mg-12Li-3Al-1.1Nd alloy. Figure 5(a) shows that, compared with Fig. 3(a), the change in number of granular Al_2Nd phase is smaller, but the size is larger. However, the needle-like $\text{Al}_{11}\text{Nd}_3$ phase is smaller and the number is much less. In the red circle of Fig. 5(b), there are some obvious holes at grain boundary, and the gray precipitated phases in the matrix significantly increase. Figure 6 shows the XRD result of Mg-12Li-3Al-1.1Nd alloy, which indicates that the matrix is single phase $\beta(\text{Li})$, and there are Al_2Nd intermetallic compounds and some MgLiAl_2 and Al_4Li_9 . Compared with Fig. 4, both the intensity and the amount of the peaks of the second phases in Fig. 6 increase significantly. Moreover, Al_4Li_9 increases and AlLi disappears.

Fig. 5: Microstructure of Mg-12Li-3Al-1.1Nd alloy at low magnification (a) and high magnification (b)

Fig. 6: X-ray diffraction pattern of Mg-12Li-3Al-1.1Nd alloy

Fig. 7: SEM micrograph (a) and area distribution of Nd element (b) in Mg-12Li-3Al-1.1Nd alloy

Fig. 8: Microstructure of Mg-12Li-3Al-2.0Nd alloy (a) and grain boundary (b)

and their vicinity, almost no needle-like $\text{Al}_{11}\text{Nd}_3$ phase appeared. In Fig. 8(b), there were a few voids at the grain boundary. The results of X-ray diffraction of Mg-12Li-3Al-2.0Nd alloy (Fig. 9) show that there were Al_2Nd intermetallic compounds and MgLiAl_2 and Al_4Li_9 on $\beta(\text{Li})$ matrix; however, the peaks of MgLiAl_2 and Al_4Li_9 declined, indicating these second phases became fewer.

As described above, the added Al can form AlLi , Al_4Li_9 and MgLiAl_2 phases with Li and Mg. But if Nd is added into the Mg-12Li-3Al alloy, due to the larger difference in electronegativity between Al and Nd (Al: 1.61; Nd:1.20), then Al is easier than Mg (Mg:1.31) to combine with Nd to

Figure 7 shows the SEM micrograph and area distribution of Nd element in Mg-12Li-3Al-1.1Nd alloy. It was evident that Nd element was enriched in the white particles and white needle-like phases, which further indicates that these white particles and the white needle-like phases are Al_2Nd and $\text{Al}_{11}\text{Nd}_3$ respectively. Al_2Nd particles were located in the matrix near the grain boundary, but not at the grain boundary, and they could hinder the long-range diffusion of other elements in the aging process.

Figure 8 is the SEM micrograph of Mg-12Li-3Al-2.0Nd. It can be seen from Fig. 8(a) that a large amount of white Al_2Nd particles was generated, and aggregated at the grain boundaries

Fig. 9: X-ray diffraction pattern of Mg-12Li-3Al-2.0 Nd alloys

form a compound with higher stability^[9,17]. Therefore, Al and Nd formed Al_2Nd and $\text{Al}_{11}\text{Nd}_3$ phase in this study, which has a precipitation strengthening effect. Meanwhile, some Al dissolved into the matrix, which also has the solid solution strengthening effect. With the increase of Nd content, the Al-Nd intermetallic compounds had a trend to change from needle-like $\text{Al}_{11}\text{Nd}_3$ to granular Al_2Nd . Al_2Nd and $\text{Al}_{11}\text{Nd}_3$ distributed near the grain boundary. But when the content of Nd was too high (e.g. 2.0wt.%), the granular Al_2Nd gathered into clusters in the vicinity of the grain boundary as well as at the grain boundary. In addition, with the increase of Nd content, Al_4Li_9 increased and AlLi disappeared.

2.3 Mechanical properties

2.3.1 Microhardness

Figure 10 shows the micro-hardness of as-cast Mg-12Li-3Al-xNd alloy in the natural aging process. It appeared that the micro-hardness of alloy after three days was basically the same as that of alloy after six months, which indicated that the addition of Nd can effectively improve the stability of the alloy at room temperature. With the increase of Nd content, the micro-hardness of the alloy increased firstly and then decreased, and reached the peak value of 75 HV at 1.1wt.%Nd. What was necessary to point out was that the poor stability at room temperature was a significant drawback of Mg-Li alloy with single phase β , e.g. LA141A^[18,19].

Fig. 10: Micro-hardness of Mg-12Li-3Al-xNd (x= 0.3, 0.7, 1.1, 2.0wt.%) alloys

2.3.2 Tensile property and fracture analysis

(a) Tensile properties of alloy

Table 2 shows the tensile properties of the alloys. It appeared that, with the increase of Nd content, the tensile strength of the alloys decreased firstly then increased, and finally decreased. The tensile strength of #3 alloy was the highest (180 MPa), but its elongation was only 9.2%; while the tensile strength of #2 alloy was only 147 MPa, but its elongation was as high as 53.7%. The reasons were as follows: While it should be pointed out that the refining effect of rare earth elements on Mg-Li alloy has been proposed in some previous literatures, the analysis of

the mechanism was discussed mainly from the point of view of second phases and solution strengthening in this paper.

For #2 alloy, the actual Al content was relatively low (as in Table 1), resulting in the smaller solid solution strengthening effect of Al. Although 0.7wt.%Nd element was added, its solubility was very small. Owing to the addition of Nd%, granular Al_2Nd and acicular $\text{Al}_{11}\text{Nd}_3$ intermetallic compounds formed, which further weakened the effect of solid solution strengthening of Al^[7]. Moreover, if compared with the remaining samples, the amount of precipitated phases in the matrix of #2 alloy was obviously less, which also resulted in the lower precipitation strengthening effect. Although the generated Al_2Nd and $\text{Al}_{11}\text{Nd}_3$ have dispersion strengthening effect, their amount was small, hence their enhanced effect was limited. Therefore, #2 alloy has the lowest tensile strength. Nevertheless, the tensile strength of #2 alloy was still better than LA141A(T7). In addition, because there was almost no shrinkage hole in the microstructure, #2 alloy exhibited excellent elongation.

In comparison, the intermetallic compound of Al-Nd formed in #3 alloy was mostly granular Al_2Nd instead of acicular $\text{Al}_{11}\text{Nd}_3$, dispersed near the grain boundaries. Compared to the acicular $\text{Al}_{11}\text{Nd}_3$, granular Al_2Nd has smaller fragmentation effect to the matrix, and can play a better role in pinning the dislocation in the process of tensile deformation. Moreover, MgLiAl_2 and Al_4Li_9 particles in the matrix also increased significantly. These particles have good pinning effect to dislocation in the process of tensile deformation too, so that the tensile strength of #3 alloy was the highest. However, these particles above decreased the ductility of the alloy, and due to the existence of some obvious shrinkage at the grain boundary, the elongation of #3 alloy was the lowest.

Compared with #3 alloy, the solid solution strengthening effect of #4 alloy was slightly weaker due to the smaller Al content, and the number of precipitated phase in matrix was smaller, so the tensile strength of #4 alloy was lower than that of #3 alloy, and the shrinkage led to the lower elongation than that of #2 alloy. Even so, the comprehensive mechanical properties of #4 alloy were still the best, and the tensile strength and elongation of #4 alloy were better than that of LA141A (T7) as well.

Table 2: Mechanical properties of Mg-12Li-3Al-xNd alloys

Number of alloy	Alloy	TS R_m (MPa)	EL A(%)	Notes
#1	Mg-12Li-3Al-0.3Nd	163	23.7	
#2	Mg-12Li-3Al-0.7Nd	147	53.7	
#3	Mg-12Li-3Al-1.1Nd	180	9.20	Shrinkage
#4	Mg-12Li-3Al-2.0Nd	169	32.5	Shrinkage
	LA141A (T7) ^[7]	144.69	24	

(b) PLC phenomenon of alloy at room temperature

Figure 11 shows the stress-strain curves of the four alloys, which indicated that the PLC (Portevin-Le Chatelier^[20]) phenomenon occurred during the tensile process at room temperature, and the PLC effect of #2 and #4 samples were especially remarkable. The reasons were as follows:

In the early stage of deformation, the beginning of dislocations in the grains must overcome the bound of solid solution atoms in the lattice. When the applied shear stress reached a certain value to free the dislocations, plastic deformation began, so the alloy exhibited obvious yield phenomenon. Because of the addition of Nd element, rare earth intermetallic compounds Al_2Nd or $Al_{11}Nd_3$ were generated in the vicinity of grain boundaries. Moreover, there were some precipitates and solid solution atoms in the matrix near the grain boundaries as well. Al_2Nd precipitates and solid solution atoms had a pinning effect for the dislocations. In the tensile process, the dislocations moved to the area near the grain boundary and piled up, resulting in a strong interaction between Al_2Nd , precipitates, solid solution atoms and the dislocation. It required a great external shear stress to free the dislocation from the bound of the solute atom group, Al_2Nd or $Al_{11}Nd_3$ and precipitated phase. Once the dislocation was free, it moved into the adjacent grain boundaries under a much lower stress, and offset with the opposite sign dislocations, resulting in the annihilation of dislocations. This process led to a stress fluctuation, and thus caused the PLC phenomenon^[21]. For #3 alloy sample, due to the evident defects, fracture occurred in the tensile process before the pile up of a large number of dislocations had been produced in the vicinity of grain boundaries, so the stress-strain curves of the #3 alloy samples did not show an obvious PLC phenomenon.

(c) Fracture analysis of alloy

The crystal structure of $\alpha(Mg)$ is hexagonal close-packed (hcp), and has only one group of slip surface (0001). When added with Li and Al alloy, the matrix of Mg-Li transformed from

$\alpha(Mg)$ into $\beta(Li)$, whose glide planes were (112) and (110). The increasing of slip system made the slip more easily, so the ductility of the alloy increased. After the tensile test, obvious necking can be observed on the fracture specimens except for #3 alloy.

Figure 12 shows the tensile fracture morphology of the four alloys. There were many equiaxed dimples on the fracture of #1 and #2 alloys which showed obvious ductile fracture characteristics, and there was a small amount of cleavage torn edges or small cleavage steps which indicated a few characteristics of quasi cleavage fracture. The fracture of #3 alloy has some tongue patterns but few dimples, and due to defect of shrinkage, the bright grain interface and the crystal grains can be seen in the defect area. Therefore, the fracture mode of #3 alloy was mainly intergranular brittle fracture.

The tensile fracture of #4 alloy also had many dimples, where a large number of small second phase particles distributed. This indicated that the second phase particles were the nucleation point of the micro-hole, whose growth and separation led to the formation of dimples. At the same time, a few tearing edges and tongue patterns could be observed. Therefore, the fracture modes of #4 alloy basically belonged to ductile fracture, accompanied by a little quasi cleavage fracture.

It was necessary to point out that there were some obvious deformed holes on the fractures of #1, #2 and #4 specimens. This phenomenon was especially remarkable on #2 sample, where the adjacent holes connected with each other, and then became a deep groove during the deformation process. The existence of deformed holes implied the plastic deformation of the alloy was adequate.

3 Conclusions

(1) Mg-12Li-3Al-xNd ($x=0.3, 0.7, 1.1, 2.0\text{wt.}\%$) alloys were prepared, whose matrix was β phase structure with Al_4Li_9 , $MgLiAl_2$, $AlLi$ phase distributing on it. With the increase of Nd content, the Al-Nd intermetallic compounds changed from needle-like $Al_{11}Nd_3$ to granular Al_2Nd , distributing in the vicinity of the grain boundaries.

(2) The tensile strength of Mg-12Li-3Al-1.1Nd was as high as 180 MPa, the elongation rate of Mg-12Li-3Al-0.7Nd reached 53.7%, and the comprehensive mechanical properties of Mg-12Li-3Al-2.0Nd was the best.

(3) PLC phenomenon occurred during tensile process of the alloys at room temperature.

Therefore, the β -based Mg-Li alloy with good plasticity as well as enhanced strength can be obtained by moderate addition of Nd and Al.

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Fig. 11: Stress-strain curves of Mg-12Li-3Al-xNd ($x=0.3, 0.7, 1.1, 2.0\text{wt.}\%$) alloys

Fig. 12: Tensile fracture of Mg-12Li-3Al-xNd alloy: (a) $x=0.3$, (b) $x=0.7$, (c) $x=1.1$, (d) $x=2.0$

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