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Materials

Design of an age hardening Mg-Li alloy and its aging behavior

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Abstract: An effort was made to design an age hardening Mg-Li alloy based on the phase diagrams calculated by means of "THERMO-CALC"—a thermodynamic software. Experiments were carried out to verify the calculated results by melting the alloy and examining its structure and aging behavior. The results show that the alloy possesses a structure constituent as expected, besides, the alloy has apparent aging behavior and over aging happens even at lower temperature. Metastable (Mg, Li, Al, Zn) phase has been identified when the hardness reaches the aging peak. With the increase of the aging time, (Mg, Li, Al, Zn) phase transforms to stable α phase and over aging happens.

Key words: Mg-Li alloy; composition design; thermodynamic calculations; aging

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1 Introduction

Magnesium alloys have been considered as one of most potential advanced structural materials because of its low density (1/3 lower than aluminum), high specific strength and specific stiffness [1]. So magnesium alloys have been found wide application in airplane and automobile industries [1-3]. However, the poor ductility of magnesium makes it difficult to forming because of its hcp structure. And magnesium alloys normally have low strength and poor precipitation hardening effect. All those confine its application field.

Some researches have shown that adding lithium into magnesium could not only lower the density, but also bring about the transformation of α(hcp)→ α+β→ β(bcc) [1,4]. With the increase of β(bcc) phase in the matrix, magnesium alloys' deformability would be enhanced apparently. According to this characteristic of Li, a series of new Mg-Li alloys, which are the lightest to normal magnesium alloys and known as super light alloys with a density of 1.35-1.65 g/cm^3, have been developed [5-7]. However, the strength of new Mg-Li alloys decreases observably with the increase of β phase in the matrix. So how to develop a Mg-Li alloy with low density and good plasticity and moderate strength becomes a critical issue.

To resolve this problem, third alloying elements such as Al, Zn and Ag were added. Those elements have an obvious solution strengthening effect on the matrix [8]. The second phases would precipitate from the matrix with Al and Zn alloying elements after appropriate aging treatment [8]. However, to the quaternary Mg-Li-Al-Zn alloy, it is time and labor consuming to design and optimize its composition for structure constituent and mechanical properties expected by means of experiment.

In order to save the experimental time and cost, a thermodynamic software "THERMO-CALC" was employed. This paper designed a new Mg-Li-Al-Zn alloy based on the phase diagrams calculated by the "THERMO-CALC". Meanwhile, experiments were carried out to verify the feasibility and reliability of the thermodynamic calculations, and to study the aging behavior of this new Mg-Li alloy. The work will supply a method and data to the R&D of advanced magnesium alloys.

2 Composition design

In order to obtain a new alloy with good ductility, the Li content of the new alloy was selected at 12wt%. As shown in **figure 1,** Mg-12wt%Li alloy will wholly transit to P(bcc) by suitable solution and quenching treatment. Meanwhile, proper contents of Al and Zn alloying elements were added to generate aging strengthening. The principle of composition design is

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that Al and Zn contents in the alloy should approach their solubility limits in the matrix for gaining high strengthening effect as possible, and the new alloy should keep the β(bcc) matrix.

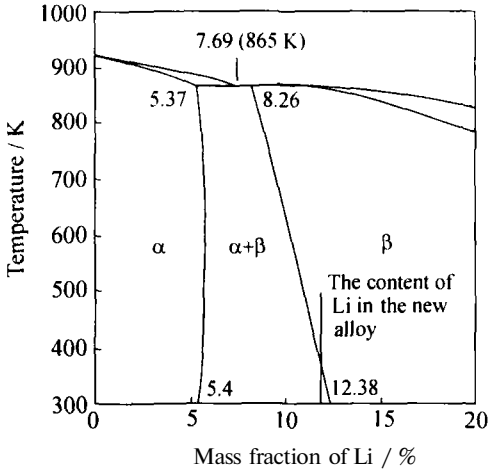

Figure 1 Phase diagram of the Mg-Li alloy [9].

To confirm an appropriate addition of Al and Zn, the changes of the Mg-Li binary phase diagram caused by introducing those elements should be considered. The key sections of the Mg-Li-Al and Mg-Li-Al-Zn alloy's phase diagrams are calculated and plotted in this paper. **Figure 2** is the vertical section of the phase diagram of the Mg-12wt%Li-Al alloy. It shows that the content of Al should be considered as about 3wt%. For the reason that the expected aging strengthening effect will reduce if the content of Al lower than 3wt%, while the alloy matrix will not be the single β(bcc) phase after solution and quenching treatment if the content of Al higher than 3wt%. **Figure** 3 is the vertical section of the phase diagram of the Mg-12wt%Li-3wt%Al-Zn alloy. As shown in Figure 3, the temperature region of single β phase existing becomes narrower with the increase of the content of Zn in the alloy. It means solution treatment operation becomes difficult. At the same time, it should also be considered that the increase of Zn in the alloy will cause the density increase of the Mg-Li alloy, which is disadvantage for the Mg-Li alloy to keep its low density.

By overall consideration, the nominal composition of the new alloy was confirmed as Mg-12wt%Li-3wt%Al-5wt%Zn. As shown in figure 3, it can be expected that the matrix of the new alloy will transit to β phase wholly after the solution and quenching treatment and some amount of AlLi and Mg_2Zn_3 phases may precipitate from the β phase matrix after appropriate aging treatment. Then the detectable precipitation hardening effect of precipitation phases can be measured by examining the aging behavior of the new alloy.

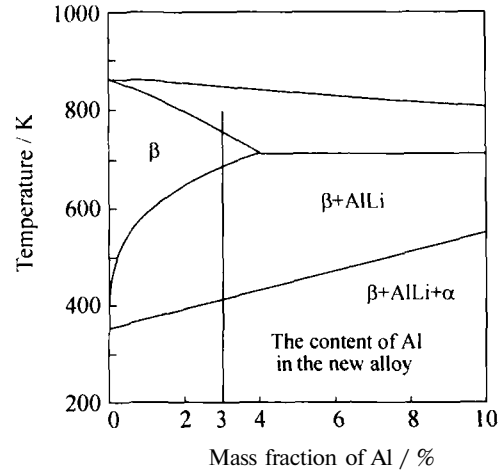

Figure 2 Vertical section of the Mg-12wt%Li-Al alloy's phase diagram.

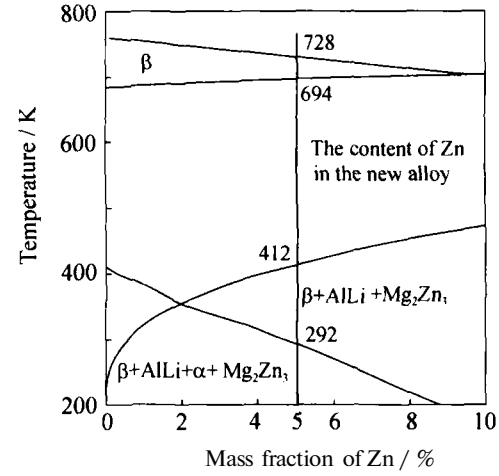

Figure 3 Vertical section of the Mg-12wt%Li-3wt%Al-Zn alloy's phase diagram.

3 Experimental details

3.1 Alloy preparation

In order to keep the smelting from oxidation, the smelting was carried out in a high pure argon atmosphere. Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) was used to analyze the composition of the ingot. The actual composition (wt%) of the Mg-Li-Al-Zn alloy is Li, 10.8; Al, 3.44; Zn, 4.96; Mg, balance.

The homogenizing treatment was carried out at 493 K for 12 h, then at 693 K for 12 h, and the alloy was hot forged into a ϕ20 mm bar at 523 K for eliminating the casting defects and making an improvement in the microstructure.

3.2 Heat treatment and hardness testing

Considering the effect of the composition shift from the nominal alloy to ingot, the vertical section of the phase diagram corresponding to the obtained alloy was also calculated and plotted **(figure 4).** Comparing figure 4 with figure 3, the fluctuation of Li and Al

content makes some changes of the existing condition of the single β phase region. Especially, the precipitation temperature of secondary α phase ascends obviously. It means that the experimental alloy may precipitate some amounts of secondary α phase besides AlLi and Mg_2Zn_3 phases after solution and aging treatment.

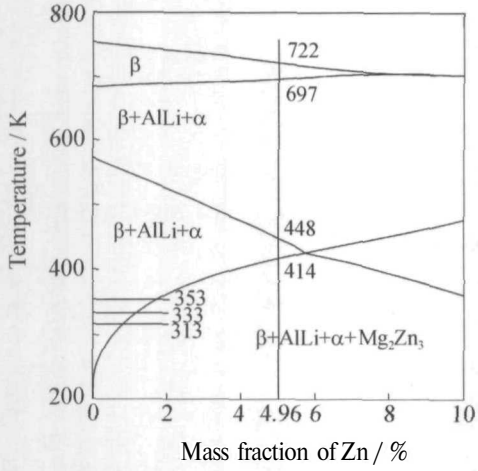

Figure 4 Vertical section of the experimental alloy's phase diagram.

As shown in figure 4, the temperature range of the single β phase region of the experimental alloy is 697-722 K, so the samples were solution treated at 713 K for 1 h. In order to keep the Mg-Li-Al-Zn alloy from oxidation, specimens (ϕ8 mm×5 mm) were solution treated in a nitrogen atmosphere **(figure 5),** then quenched into the water at room temperature.

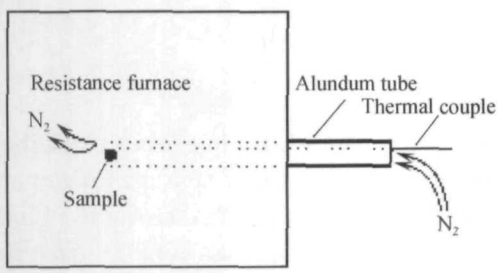

Figure 5 Facilities used for solid solution treatment.

Aging tests were carried out at 313, 333 and 353 K respectively in a silicone oil bath. In order to study the aging behavior of the Mg-Li-Al-Zn alloy, a Vickers hardness tester (HV-0.5) was used. The load employed was 9.8 N and maintained for 30 s.

3.3 Microstructure observation

The sample microstructure was examined by using an optical microscope. All the samples used for microstructure observation were ground and polished and then etched with nitric acid alcohol. The precipitate of the alloy was determined by X-ray diffraction techniques.

4 Results and discussion

4.1 Solution treatment

As shown in **figure** 6, second phases were not observed in the matrix grains and the grain boundaries basically. As shown in **figure 7,** the XRD pattern of the experimental alloy further indicates that the alloy matrix is single β phase as expected after solution treatment. So the solution treatment parameters (at 713 K for 1 h) confirmed by the results of thermodynamic calculation are feasible.

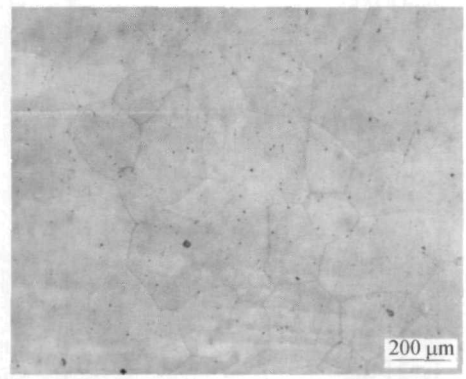

Figure 6 Optical microstructure of the solution treated sample.

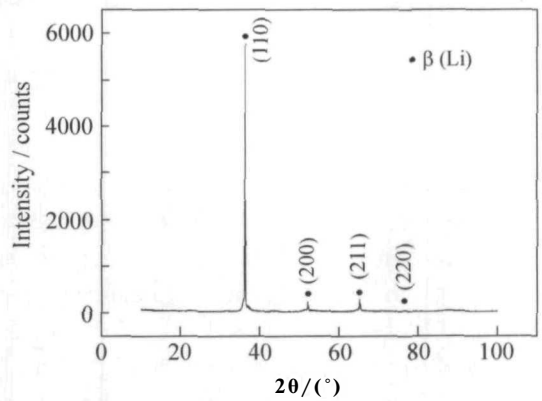

Figure 7 XRD pattern of the solution treated sample.

4.2 Hardness curves

Figure 8 is the hardness dependence of aging time at 313, 333 and 353 K respectively for the alloy. It shows that the peak hardness of the alloy aged at 313

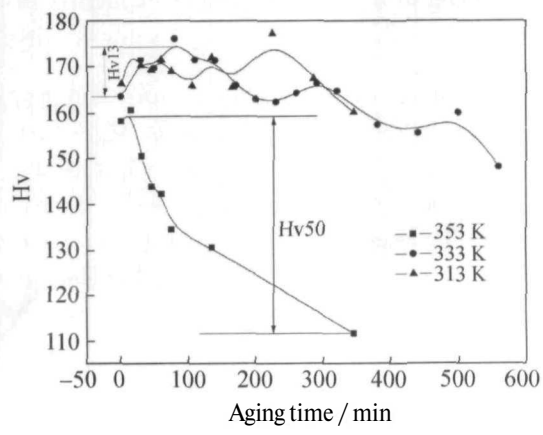

Figure 8 Aging curves of the experimental alloy.

K for 225 min increases to about Hv 177. And with the increase of the aging temperature, the hardness peak appears sooner. The hardness of the alloy aged at 333 K reaches its peak (Hv 176) for 80 min. While, the hardness of the alloy aged at 353 K decreases rapidly without obvious hardness peaks.

Comparing the hardness of the peak aging treated samples at 313 and 333 K with that of the solution-treated samples, the increase in hardness by age hardening is less than 10% of the latter's hardness. The age hardening effect is relatively small.

4.3 Precipitation mechanism and XRD analysis

Figures 9 and **10** are the XRD patterns of the aging treated samples at 333 K for 80 and 350 min, respectively. As shown in figure 9, after aging at 333 K for 80 min, the diffraction peaks of (Mg, Li, Al, Zn) phase (JCPDS card file No.17-919) which has the fcc structure are identified. And after aging at 333 K for 350 min, the diffraction peaks of α are identified. Comparing figures 9 and 10 with 8, it can be confirmed that the strengthening phase is (Mg, Li, Al, Zn) phase, and with the increase of the aging time, (Mg, Li, Al, Zn) phase transforms to α phase and over aging happens.

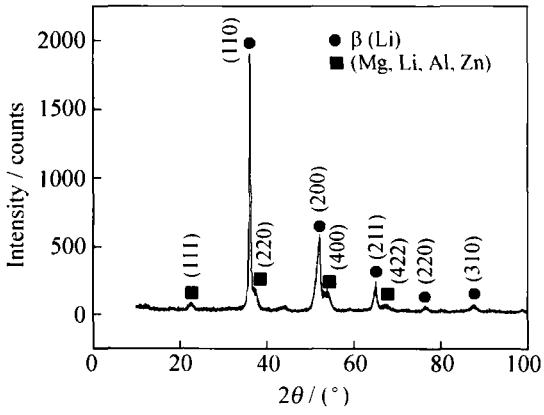

Figure 9 XRD pattern of the sample aged at 333 K for 80 min.

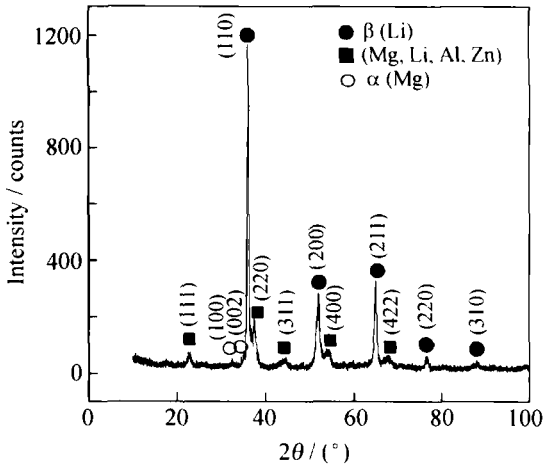

Figure 10 XRD pattern of the sample aged at 333 K for 350 min.

Figure 11 is the XRD pattern of the over-aging sample at 353 K. In this figure, the diffraction peaks of (Mg, Li, Al, Zn) and α phase are identified.

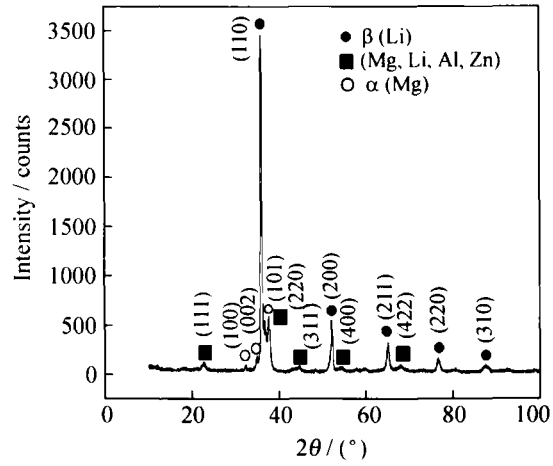

Figure 11 XRD pattern of the sample aged at 353 K for 171 min.

From the aging curves and phase analysis we could make the prediction that (Mg, Li, Al, Zn) phase is a transition phase to α, its composition (wt%) is: Mg, 84.2, Li, 12.8; Al, 1.33, Zn, 0.92 [10]. So aged at the above temperature, metastable (Mg, Li, Al, Zn) phase which has similar chemical constitution with β matrix precipitates from the matrix, firstly, and the hardness of the alloy increases until the hardness peak. With the increase of the aging time, (Mg, Li, Al, Zn)-fcc structure phase transforms to stable α-hcp structure phase which has similar crystal structure with (Mg, Li, Al, Zn) phase, and over aging happens.

The diffraction peaks of AlLi and Mg_2Zn_3 did not appear aged at the above described temperature. Maybe those two phases would precipitate at a higher temperature or a longer aging time. This should be verified by further experiments. Because aging is a dynamic course, while the phase diagrams calculated by "THERMO-CALC" are equilibrium diagrams, so those phase-diagrams can only instruct the aging treatment to some extent.

5 Conclusions

(1) It is beneficial to design a new Mg-Li alloy and confirm the solution and aging treatment parameters by means of thermodynamic calculations.

(2) The age hardening effect of the experimental alloy is not satisfied, though the (Mg, Li, Al, Zn) phase is identified when the alloy reaches its peak hardness. With the increase of the aging time, unstable (Mg, Li, Al, Zn) phase transforms to stable phase α, and overage occurs. With the increase of the aging temperature, the hardness peak appears sooner. The

heat stability of the experimental alloy is relatively bad. The hardness of the alloy aged at 353 K decreases rapidly. The strengthening effect and heat stability of the experimental alloy need to be improved.

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