Effect of Solution Treatment on Microstructure and Properties of Gd - AZ91 Magnesium Alloy

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Abstract. In this paper, the Gd-AZ91 alloy was manufactured by adding rare earth element Gd in AZ91 magnesium alloy. The effects of solution treatment on the microstructures of rare earth elements Gd were investigated by means of optical microscopy, scanning electron microscopy, X-ray diffraction analysis and equipment for testing mechanical properties. The experimental results show that the addition of rare earth element Gd in AZ91 magnesium alloy can refine the alloy grain, turn β-Mg17Al12 phase into a discontinuous network or point structure, and produce granular compound Al2Gd in the alloy; when solution temperature is about 380 °C, the alloy structure is the best, the tensile strength of the alloy is the largest with the value larger than 250Mpa; when the solution temperature exceeds 380 °C, the alloy structure is coarsened and the mechanical properties of the alloy are reduced. With the increase of rare earth element Gd content, the tensile strength of the alloy shows a tendency to increase gradually, which Indicates that the addition of a certain amount of rare earth elements Gd can improve the plasticity of the alloy.

1. Introduction
Magnesium alloy is the lightest metal structure material in practical application. It has many excellent properties such as high specific strength and specific rigidity, good cutting performance, ductility and casting performance. And also, it has high dimensional stability, the buffer capacity, thermal conductivity and good conductivity. Magnesium alloy processing energy consumption is low and the pollution to the environment is small [1-4], which makes magnesium alloy materials very popular in the fields of communications, computer, aerospace, automotive, and medical devices. AZ91 magnesium alloy is Mg-Al-Zn-Mn alloy which the most widely used one. It has good casting performance and high yield strength, which can be used for various forms of mechanical parts. So it is increasingly widely used in aerospace and automotive industries [5]. But there are still some technical difficulties restricting the application of magnesium alloy parts. For example, the strength of magnesium alloy parts is not high, its high temperature strength weak, its corrosion resistance low and creep resistance limited, which restrict its application to important structural parts and greatly limit the high-end application and promotion of this magnesium alloy. However, to improve the mechanical properties of AZ-based magnesium alloy has a variety of solutions, such as alloying to improve the alloy structure, heat treatment strengthening and deformation strengthening, fine grain strengthening, and dispersion strengthening [6]. Therefore, for the above problems, this paper discusses the effect of solution treatment on the microstructure and mechanical properties of AZ91 magnesium alloy containing rare earth Gd in industrial application and its extensive suppression of magnesium alloy AZ91.
2. Test Materials and Methods

The test materials are AZ91 magnesium alloy, which is commercially available, it contains: Ag 9.1%, Zn 0.93%, Mn 0.36%, Si≤0.02, Fe≤0.12, other impurity elements less than 0.04%, and the rest is Mg. With the mixed refrigerant YJ134a and nitrogen as a protective gas, the magnesium alloy was smelted in the 8GWU resistance furnace. When the temperature is during 690 °C ~ 720 °C, add different proportions of Mg-Gd rare earth master alloy, and stir the magnesium alloy melt and keep temperature for 10 minutes. And then prepare AZ91-Gd magnesium alloy experimental samples for the test in J1125B die casting machine. The die-casting temperature is during 680 °C ~ 710 °C. The rare earth elements Gd in the alloy is shown in Table 1. The new Gd-AZ91 alloy was manufactured. The die-casting sample size and die-casting process are designed accordance to the national standards GB / T13822-92 for die-casting alloy.

Table 1. Gd component design of rare earth elements in AZ91 alloy (mass fraction, %)

| Alloy Element / wt.% | Al   | Zn   | Mn   | Gd | Mg   |
|----------------------|------|------|------|----|------|
| A0                   | 9.0  | 0.8  | 0.2  | 0  | Bal  |
| A1                   | 9.0  | 0.8  | 0.2  | 0.2| Bal. |
| A2                   | 9.0  | 0.8  | 0.2  | 0.4| Bal. |
| A3                   | 9.0  | 0.8  | 0.2  | 0.6| Bal. |
| A4                   | 9.0  | 0.8  | 0.2  | 0.8| Bal. |
| A5                   | 9.0  | 0.8  | 0.2  | 1.0| Bal. |

3. Experimental Results and Discussion

According to the heat treatment scheme, Figure 1 (a) shows the tensile properties of the alloy under different temperature holding conditions at 380 °C solid solution temperature. Figure 1 (b) shows the tensile properties of the alloy at different solution temperatures.

(a) Different solution time; (b) different solution temperature;

Figure 1. Effect of solution temperature and time on tensile strength of alloy

According to Figure 1 (a), when T = 380 °C, and prolonging the holding time, the tensile strength of the alloy increases first and then decreases. When t = 12h, the tensile strength of the alloy is the largest with the value larger than 250MPa. When t = 16h, the tensile strength of the alloys decreased. According to Figure 1 (b), when T = 380 °C and changing solution temperature, the tensile strength of the three alloys increased first and then decreased, the tensile strength of the alloy is the maximum.

Figure 2 shows the optical microstructure of the alloy containing rare earth 0.6% Gd when the holding time is 8h, 10h, 12h or 16h after solution treatment at 380 °C. It can be observed from Figure 2 (a) to (c) that the coarse network β-Mg17Al12 phase is tapering and the content decreases with the
increase of the solution time. When \( t = 16 \) h (Figure 2 (d), a discontinuous network structure appeared. According to the literature [7], the Al\(_2\)Gd phase was changed from needle-like, rod-like, short rod-like to granular in the alloy.

Figure 2. Microstructure of 0.6\%Gd alloy with different period at 400 °C

Figure 3 shows the microstructure of the alloy containing rare earth 0.6\% Gd after the holding time of 12 h at different solution temperatures. It can be seen from Figure 3 (a) that a large number of finer mesh \( \beta \) phase and a small amount of larger particles can be seen at 360 °C. When the solution temperature is at 380 °C (as shown in Figure 2 (c)), alloy refinement can be easily observed, and the particles become smaller in diameter. Also, the number of the particles increases and distribution looks more uniform. But when \( T = 410 \) °C (as shown in Figure 3 (b)), the surface of the sample appears to crack and melt, that is the typical over-burning (as shown by the red arrow in Figure b) which results in a rapid drop in the strength of the alloy.

Figure 3. Microstructure of 0.6Gd alloy with the holding time of 12h at different solution temperature
In order to analyze the change of the phase after the solution treatment, XRD diffraction analysis was performed on the alloy treated with the solution containing rare earth 0.6% Gd. The analytical results are shown in Figure 4.

![XRD diffraction pattern of A3 alloy](image)

**Figure 4.** XRD diffraction pattern of A3 alloy

It can be seen from Fig.4 that the solid solution alloy is mainly composed of $\alpha$-Mg phase, $\text{Al}_2\text{Gd}$ phase and a small amount of $\beta$-$\text{Mg}_{17}\text{Al}_{12}$ phase, and the peak of $\beta$ phase is obviously reduced, which indicates that after the alloy containing rare earth 0.6% Gd is treated with solid solution, the $\beta$-$\text{Mg}_{17}\text{Al}_{12}$ phase in the alloy is basically dissolved. However, the rare earth compound $\text{Al}_2\text{Gd}$ is not dissolved after the solution treatment at 380 °C because of its high melting point. Similarly, calculate the different phases in the alloy containing rare earth 0.6% Gd with 380 °C×12h solution treatment through the RIR method. After alloy containing rare earth 0.6% Gd after solution treatment: $R = 2.93$, wt (Mg) % = 90.526%; wt ($\beta$-$\text{Mg}_{17}\text{Al}_{12}$) % = 0.212%. It can be seen that the solution-treated $\beta$-$\text{Mg}_{17}\text{Al}_{12}$ phase is basically dissolved, while the rare earth phase $\text{Al}_2\text{Gd}$ still exists.

In order to observe the change of the microstructure of the alloy after cleansing treatment, the rare earth magnesium alloy with different content was analyzed by transmission electron microscopy. It can be seen from Figure 5 that after solution treatment, the precipitates in the alloy become more refined and distribute dispersive.
According to the Al-Gd phase diagram and the Mg-Al phase diagram, the Al$_2$Gd phase is stable (its melting point can reach 1525 °C), the Al$_2$Gd phase is stable, while the thermal stability of β-Mg$_{17}$Al$_{12}$ phase is poor, and it decomposes at about 300 °C. Therefore, after the solution treatment at 380 °C, the rare earth phase Al$_2$Gd produced by the alloy in the as-cast state can exist stably, but the β-Mg$_{17}$Al$_{12}$ phase decomposes. Thus, the reticular β phase in the alloy after the solution treatment is continuously reduced, and the particulate matter or the bulk Al$_2$Gd still exists.

In the alloy with different solution time, at some temperature, the β-Mg$_{17}$Al$_{12}$ phase is the strengthening phase in the AZ91 magnesium alloy and is characterized with high hardness. Because of the addition of the rare earth element Gd when solution treatment is done, some of β-Mg$_{17}$Al$_{12}$ combined with Al, and some dissolved in the α-Mg matrix or replaced the Mg in β-Mg$_{17}$Al$_{12}$ phase to produce (Mg, RE)$_{17}$Al$_{12}$ phase. So, the addition of rare earth can pin the diffusion of Al, increase the concentration of Al around (Mg, RE)$_{17}$Al$_{12}$ phase, and decrease the kinetic condition of its decomposition, which slows down the decomposition of β phase.

4. Conclusion
1) The tensile strength of AZ91 magnesium alloy containing Gd was improved by solution treatment. When the solution treatment was: 380 °C×12h, the alloy structure was the finest, the distribution of precipitates is the most uniform, and alloy tensile strength is the largest, with the value larger than 250Mpa. When exceeding 380 °C, the organization began to coarser and mechanical properties decreased. When reaching 410 °C, it was over-burning.

2) The β phase in the alloy, which is mainly composed of α-Mg phase, Al$_2$Gd phase and massive Al-Mn phase, is basically decomposed after the solution treatment at 380 °C×12h. With the increase of Gd content, the size of the alloy crystal becomes smaller, the number of Al$_2$Gd phase increases. When the Gd content is 1.0%, the grain structure of the alloy is improved significantly.

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