Effect of Gd on Microstructure and Fracture Behavior of AZ91 Magnesium Alloy

Yao Li*a, Junjie Yang and Huiling Wang
School of Electromechanical & Architectural Engineering, Jianghan University, Wuhan, Hubei, China, 430056
Email: *lylly111@sina.com

Abstract. Alloy was prepared by adding Rare Earth (RE) elements Gd in AZ91 Magnesium alloy. With Optical Microscope, Scanning Electron Microscope, X-ray diffraction analysis and mechanical properties testing equipment, the paper explored the influence of RE element Gd with different content on microstructure and fracture behavior of as-cast AZ91 Magnesium alloy. The results show that the addition of RE element Gd in Magnesium alloy AZ91 can refine grain, make β-Mg17Al12 phase become a discontinuous network or punctate structure, and generate granular particulate Al2Gd in the alloy. With the increase of the content of Gd, tensile strength, elongation and hardness of the alloy showed a gradually increasing trend. The tensile fracture of AZ91 magnesium alloy at room temperature was obvious tearing pattern of river pattern, which shows that it is dominant of cleavage brittle fracture. After adding a certain amount of Gd, the plasticity of the alloy has been improved, and the dimple and tearing edges begin to appear on the fracture surface, which show the features of quasi cleavage fracture and plastic fracture.

1. Introduction
Among currently applied metal materials, magnesium alloy, the lightest metal material, possesses numerous marvelous properties, such as high specific strength, stiffness and dimensional stability, good machinability, ductility, casting property and thermal conductivity, strong ability against vibration and noise, low energy consumption and pollution in magnesium alloy processing [1]. Owning to above merits, magnesium alloy is widely used in various fields, including communication, computer, aerospace, vehicle, medical devices and so on. AZ91 magnesium alloy is a kind of Mg-Al-Zn alloys, which is the most widely used alloy, and is famous for its good casting properties and high yield strength. It can be applied in varied mechanical accessories of vehicle and aerospace [2]. However, at present, the application of magnesium alloy accessories in automobile is unexpectedly limited because of some technical difficulties, such as poor intensity under high temperature, low corrosion resistance and limited creep resistance [3]. Plenty of methods are proposed to enhance the mechanical performance of AZ magnesium alloy, like alloying to improve microstructure, heat treatment strengthening, deformation strengthening, fine grain strengthening, dispersion strengthening [4,5]. Hence, with the varying content of RE Gd adding in AZ91 magnesium alloy, the effects of RE Gd on casting microstructure and fracture properties are discussed in this paper.

2. Materials and sample preparation
The samples used in this paper were directly bought from markets, which contain 9.1% Al, 0.93% Zn, 0.36% Mn, less than 0.02% of Si, less than 0.12% of Fe, less than 0.04% of impurity and the rest Mg. The preparation process was going on in a resistor furnace (8GWU) with shielding gas including...
mixed cryogen (YJ134a) and N₂. When the temperature reached 690-720 °C, Mg-Gd master alloy of different ratio was added with constantly stirring and keeping warming for 10 min. The required AZ91-Gd samples were finally obtained in die casting machine (J1125B) in 680-710 °C, and the size and casting technique of samples were designed to meet the national standard (GB/T13822-92). The added Gd with different concentrations is presented in Table 1.

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

| Alloy | Element / wt.% | Al | Zn | Mn | Gd | Mg |
|-------|----------------|----|----|----|----|----|
| 1     |                | 9.0| 0.8| 0.2| 0  | Rest |
| 2     |                | 9.0| 0.8| 0.2| 0.4| Bal. |
| 3     |                | 9.0| 0.8| 0.2| 0.6| Rest |
| 4     |                | 9.0| 0.8| 0.2| 0.8| Rest |
| 5     |                | 9.0| 0.8| 0.2| 1.0| Rest |
| 6     |                | 9.0| 0.8| 0.2| 1.2| Rest |

Tensile tests were done on WDW-100 electronic universal testing machine with a speed at 2 mm/min. DMI-5000M optical microscope was used to study the optical microstructures. X'Pert Powder leaded to the analysis of chemical phases and SU8010 FE-SEM with EDS was responsible for microstructures, components and tomoscan of as-prepared AZ91. Moreover, the testing of the hardness of samples was obtained from HVS-1000A Vicker's indentation test machine.

3. Result and Discussion

According to the binary alloy phase diagram Mg-Al [6] and the ternary binary alloy phase diagram of Mg-Al-Zn [7], the casting structure of AZ91 mainly consists of α-Mg phase and β-Mg₁₇Al₁₂. In order to observe surface morphology and its changes of main phases, optical microstructures need to be carried out. The microstructures of different concentration of Gd doped magnesium alloy is shown in Fig. 1. As is illustrated in Fig. 1a, the cast AZ91 is comprised of α-Mg which presents white coarse dendrite morphology, and β-Mg₁₇Al₁₂ which lies in crystal boundary of α-Mg and possesses black mesh structure. The Fig. 1b shows that, adding 0.4% Gd, the β-Mg₁₇Al₁₂ phase is broken, and with the increase of Gd, it is shown in Fig. 1c that the mesh structure becomes less and slenderer, the crystal particle of α-Mg is smaller and the β phase is dispersing. However, as is illustrated in Fig. 1d, non-β compounds can be seen on the crystal boundary and in the crystal when the content of Gd is 1.2%. Based on heterogeneity theory [8], in order to promote liquid metal nucleating with nucleator, high melting point phase is required, which provides heterogeneous nucleation interface and melting point of which is higher than liquid metal. In this work, the melting point of the new shaped phase--Al₂Gd, is 1525°C compared to 650°C of Mg, which means the liquid metal nucleating process is promoted. Besides, the new Al₂Gd emerges elugiation at the frontier of solid solution interface which enhances degree of super-cooling, promotes nucleation, interrupts the growth of mesh-like β-Mg₁₇Al₁₂ and makes spacing of α-Mg for the purpose of refining crystal size.

SEM and EDS is carried out to for further analysis. The results of SEM and EDS are displayed in Fig. 2 and Table 2 respectively.

In Figure 2, with the addition of Gd, consecutive and mesh-like β phase is transformed into discontinuous dots, and the size of α phase becomes smaller.
**Figure 1.** Microstructure of as cast alloy.

(a)without RE; (b)0.8% Gd; (c)1.2% Gd

**Figure 2.** SEM photos and position of EDS analysis point of cast alloy.

The result of EDS is presented in Table 2. As is shown in Figure 2a, the atomic percent of each element (Mg: Al: Zn) with mesh structure of AZ91 turns out to be 64.005:34.418:1.577. According to the ternary binary alloy phase diagram of Mg-Al-Zn, this ingredient is the phase of $\beta$-Mg$_{17}$Al$_{12}$ or eutectic structure of $\alpha$ and $\beta$, which is comprised of gray $\alpha$-Mg and a little Al.

The SEM images of AZ91 with 0.8% Gd is displayed in Fig. 2b. This kind of AZ91, shown with labels from 3 to 5, mainly consists of $\alpha$-Mg, inconstant $\beta$-Mg$_{17}$Al$_{12}$ and white particulate particles respectively. The atomic percent of each element (Mg: Al: Gd) is 17.912: 57.029: 25.059. It is deduced that the white particulate particles are Al$_2$Gd, and the Gd is distributed along the grain boundary according to a literature [9]. In addition, little Gd is found in the matrix phase of AZ91, which means Gd exists in $\alpha$-Mg.

Figure 2c is the SEM images of AZ91 with 1.2% Gd. It is clearly shown that the mesh-like of alloy is transformed into short rot structure, resulting in the smaller grain size and particles with different sizes. Table 2 contains the results of EDS, which is proposed that these particles consist of Al$_2$Gd and...
β-Mg₁₇Al₁₂, suggesting that the consecutive mesh-like β phase is first turned into inconsecutive one, then into dotlike particles. The particles results from the combination of Gd and Al. Moreover, the β phase of AZ91 with 1.2% Gd and AZ91 with 0.8% Gd in the EDS tests was found with the presence of Gd, like the label 4 and 7 in Fig. 3. Therefore, it can be known that around the β phase in crystal boundary, a new phase, Al₂Gd, is formed.

Table 2. Element content in Figure 3 (at %)

| Alloy          | Position | Mg     | Al    | Zn    | Gd   |
|----------------|----------|--------|-------|-------|------|
| Without RE     | 1        | 91.772 | 8.228 | —     | —    |
|                | 2        | 64.005 | 34.418| 1.577 | —    |
|                | 3        | 89.634 | 9.135 | —     | 1.231|
| 0.8% Gd        | 4        | 62.553 | 36.618| 0.314 | 0.415|
|                | 5        | 17.912 | 57.029| —     | 25.059|
| 1.2% Gd        | 6        | 91.454 | 5.422 | —     | 1.124|
|                | 7        | 79.716 | 14.844| 2.917 | 2.523|
|                | 8        | 13.418 | 63.005| 1.435 | 23.577|

In order to investigate the pattern of above particles and confirm the chemical composition of the rare earth phase, the TEM filed images and corresponding electron diffraction patterns were taken in further study. Figure 3a shows that the sizes of α-Mg are quite different and the β phase is mesh-like structure. The AZ91 with 0.4% Gd of light field images of TEM is displayed in Fig. 3b, in which the sizes of α-Mg become small and the black β phase is turned into inconsecutive and graininess distribution. Fig. 3 is the ones of 1.0% Gd, revealing that the decreasing black β phase and the formation of bulks in and outside the crystal as the label points out, which is able to hinder the growth of crystalline grain and the slip of grain boundary. Fig. 3d is based on the pointed bulks in Fig. 3c. According to calculation and analysis of diffraction spots in Transmission Electron Microscopy, it is concluded that the direction of electron incidence angle is [212], and the bulks should be Al₂Gd phase with face centre cubic structure [10].

Figure 3. TEM photos of alloys.
The addition of RE into alloys leads to decrease of the β phase, which results in the enhancement of strength and elongation compared to pure alloys. The enhanced mechanical properties are shown in Figure 4 and Figure 5.

Generally, the fracture of alloy is divided into fragile fracture and ductile fracture. According to the micromechanism of fracture, it consists of cleavage, quasi-cleavage, intercrystalline rupture, fatigue crack and so on. In the process of cleavage fracture, the river pattern is integrated by many sidestep-like structures. While quasi-cleavage belongs to cleavage, its extended process proceeded in a small part compared to that of cleavage, resulting in hollow at fracture surface and finally in forming numerous tears ridges. Fragile fracture is characterized with intergranular fracture and transgranular fracture along grain boundaries in polycrystals, which, on the macroscopic view, presents none yielding and necking phenomena. Ductile fracture is mainly transgranular fracture [11, 12].

Figure 6 shows SEM images of the fracture features after tensile failure. It is illustrated in Fig. 6a that the pure AZ91 possesses no pars fibrosa, low contraction percentage and smooth fracture on which few tear ridges are shown. Tear ridges of river pattern are obviously seen on the fracture with typical cleavage steps, which is mainly caused by fragile fracture. The results show that the fracture of pure AZ91 belongs to cleavage fracture under room temperature. By adding appropriate amount of Gd, as is shown in Fig. 6b, cleavage surface and tear ridges are still seen obviously in AZ91 with 0.4% Gd, which are pointed by the big arrow, but with the decrease of cleavage and increase of tear ridges, this kind of fracture can be treated as quasi cleavage. The fracture features of AZ91 with 0.8% Gd, in Fig. 6c, shows the decrease of cleavage surface and the emergence of dimples pointed by the big arrow. At the dimples, there appear tear ridges and tiny dimples, which contain compound particles. The fracture images of AZ91 with 1.2% Gd, displayed in Fig. 6d, reveals fewer tear ridges of river pattern, and more, bigger and deeper dimples pointed by small arrow, indicating the trend of plastic fracture. According to material fracture theory [12], dimples are the main micro-feature of ductile rupture. So the as-prepared alloys present some characters of ductile rupture. As a result, with adding Gd, the fracture of alloys is transformed into quasi cleavage fracture compared to cleavage fracture of pure alloys, which illustrates the rare earth element Gd plays a role in promoting the plasticity of alloys and delaying the premature fracture. This is the reason why elongation is increased after adding the rare earth element.
Figure 6. Fracture diagram.

4. Conclusion
(1) When the amount of 0.4% ~1.2% Gd is added into AZ91, the crystal can be refined, β phase is turned from consecutive mesh structure into inconsecutive distribution, and finally into particles with decreasing quantity, and the particles and bulks of Al2Gd phase are formed.
(2) With the adding of Gd, the tensile strength, elongation and hardness of AZ91 in room temperature can be also enhanced. The good mechanical properties is obtained with the concentration from 0.8% to 1.2%.
(3) The fracture of AZ91 is mainly shown as cleavage fragile fracture under room temperature. However, with the addition of Gd, the alloy plasticity is promoted, tiny dimples and tear ridges begin to emerge in the fracture morphology, showing the characters of quasi-cleavage fracture and ductile fracture.

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6. References
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