Research Article

Study on the Microstructure and Mechanical Properties of As-Extruded AZ Magnesium Alloys by Micro-Adding La Rare-Earth Element

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Changes of precipitates, dynamic recrystallization, texture, and properties at room temperature in the as-extruded AZ alloys (Al = 0, 3, 6, 8, wt.%) with 0.3% La addition were systematically investigated in this work. The analysis results suggest that new Mg12La with lamellar-structure precipitates in AZ01-0.3La alloy and new Al11La3 with needlelike structure precipitates in AZ-0.3La alloy with 3%–8% Al. Crashed Mg17Al12 could decrease the recrystallization temperature. Proper-sized Mg12La and Al11La3 could promote the dynamic recrystallization nucleation, leading to the reduction of grains in the as-extruded AZ-0.3La alloy, except for 6% Al. At room temperature, the strength of as-extruded AZ-0.3La alloys increases as Al increases, and the elongation first increases and then decreases, while the elongation is greater than 20% with 3%–8% Al. Due to the refined average grain size of 12.2 μm, the scattered and wakened (0002) base texture, the as-extruded AZ31-0.3La shows the best comprehensive tensile property compared with other AZ-0.3La alloys and AZ alloys, of which the R$_{p0.2}$ is 162 MPa, the R$_{m}$ is 252 MPa, and the A is 21.7%.

1. Introduction

Due to their low density, low cost, good damping, good mechanical properties, and easy recycling, magnesium alloys are attractive for structural materials of metal and widely used in many fields [1, 2]. Among various commercial magnesium alloys, AZ wrought magnesium alloys dominate in consumption because of their comprehensive properties, diverse shapes, and high productivity, such as AZ31, AZ61, and AZ80 alloys. However, their structural applications are still limited due to the poor formability at room temperature due to the hexagonal close-packed (HCP) crystal structure [3–5]. Furthermore, the AZ magnesium alloys containing high content of Al element have respectively low ductility because of the high volume fraction of coarse Mg17Al12 phases [6, 7].

Alloying rare-earth (RE) elements is an effective way to enhance the strength and ductility of AZ magnesium alloys, such as Gd, Nd, Pr, and Y. These rare-earth elements could react with the Al element and form Al-RE intermetallics with good thermal stability, precipitating in the matrix and affecting the initial structures of the AZ magnesium alloy, such as the Mg17Al12 phases, the grains, and the texture [8–10]. However, these RE elements are generally expensive, contributing to the relatively high cost of AZ magnesium alloys and limiting their applications in the civilian field. La belongs to the light RE and adding La to AZ magnesium alloys also affects their initial structures and properties [11–14]. In addition, La has a lower cost than Gd, Nd, and Pr, thus, AZ magnesium alloys with La additions have a lower cost than that with Gd, Nd, and Pr additions. Kumar and Gupta found that the grain structure of the as-extruded Mg-3Al-2.5La alloy could be refined by 75% compared to the pure Mg, and the R$_{p0.2}$ was 160 MPa, the R$_{m}$ was 249 MPa, and the A was 22%, and the improved properties were attributed to the dispersed formation of the Al$_6$La, Al$_{12}$La$_3$Al$_{0.6}$, and Al$_{11}$La$_3$ intermetallics [15]. Ashrafizadeh and Mahmudi studied the comparison of the effects of the separate additions of 1%Gd,
1%Y, and 1%La on the creep resistance of the AZ81 alloy, illustrating that the Al11Gd, Al12Y, and Al11La intermetallics were barriers and could limit the recovery processes. Furthermore, La had the strongest effect on decreasing the minimum creep rates [16]. Other reports also reported the positive effects of adding La or La-rich Misch metal to the AZ magnesium alloys [17–20]. However, most Mg-Al-Zn systemic alloys with La additions and well performance in the recent research have a characteristic: the La addition was more than 1% and research of microadding La is further needed.

Our previous research has shown that microadding La to AZ91 alloy could enhance the elongation at room temperature [21]. Due to the fact that Al affects the grains and mechanical properties of AZ magnesium alloys greatly, changes in microstructures and properties in Mg-Al-Zn alloys with microadditions of La and different additions of Al need to be further studied. In this work, the commercial AZ alloys (AZ61, AZ31, AZ80) were selected as the typical Mg-xAl-Zn systemic alloys, the precipitates, the DRX (dynamic recrystallization), the texture, and the mechanical properties at room temperature of AZ alloys by microadding La were studied. The purpose of the systematic investigation in this work is to make a systematic comparison of microadding La to AZ alloys, clarifying the deformation behavior and microstructure evolution, and guide the application of the promising AZ-RE alloys in the industry.

2. Materials and Methods

Commercially pure Mg, Al, Zn, and Mg-25La (wt. %) master alloys and anhydrous MnCl2 were used to prepare the studied magnesium alloys. The whole melting process was carried out in a resistance furnace under the protection of a CO2 atmosphere and flux no. 5. Firstly, the pure Mg was added to the crucible, and the crucible was heated to 700°C until the pure Mg melted. The pure Al and Zn were added to the melt and held for 5 min. After that, the temperature was raised to 730°C, and the Mg-25La master alloy was added and held for 5 min. The anhydrous MnCl2 was added to the melts at 700°C, and the melt was stirred for 2 min for full refining. Finally, the melt was held at 700°C for 10 min and cast into the steel mold under the protection of the SF6 and CO2 (the volume ratio was 1:9). The as-cast alloys were held for 10 h at 420°C and then air-cooled to room temperature. After that, the cast ingot was cut into Ø 46 mm and prepared at 350°C for 2 h before being extruded to rod bars at the same temperature. The extrusion ratio was 17.4:1 and the exit velocity was 1.4–1.5 m·min⁻¹. The technological process diagram is shown in Figure 1.

The actual composition of the materials was achieved through the ICP analysis and the results are shown in Table 1. The studied alloys were cut and ground mechanically to mirrorlike surfaces using abrasive papers and diamond pastes and characterized by X-ray diffraction (XRD) for the phase composition and texture examination, and the (0002) and (1010) pole figures were calculated with the MATLAB software. Moreover, the studied alloys with mirrorlike surfaces were etched by immersing them for 15–20 s in a solution including 10 ml acetic acid, 4.2 g picric acid, 10 ml distilled water, and 70 ml ethanol, and examined by an optical microscope (OM) and a field-emission scanning electron microscope (FE-SEM) equipped with an energy-dispersive X-ray spectrometer (EDS) system. The grain size of the recrystallized grains was calculated by the three-line method, and the average grain size was calculated by the above results. Tensile tests of the studied alloys were performed by the universal material test machine (SHIMAZU AG-IS), and the strain rate was 1.0 mm·min⁻¹.

3. Results and Discussion

3.1. Precipitates of the As-Cast Studied Alloys. The SEM images and XRD patterns of as-cast studied alloys are shown in Figure 2 for new precipitate analysis. It could be seen that white precipitates exist in all of the as-cast studied alloys, and these white precipitates are present in three morphologies: the first one is a large white particle dispersed in α-Mg matrix (marked as A, D, and G), which is Mg-Mn-La and Mg-Al-Mn-La according to the EDS analysis in Table 2; the second one is Mg12La with lamellar-structure (marked as B) according to the corresponding XRD patterns and EDS analysis in Table 2; and the third one is white Al11La3 with needlelike structure (marked as C, E, and F) according to the XRD patterns and EDS analysis in Table 2, which is rich in both Al and La with the ratio of about 11:3. That is to say, La prior reacts with Al compared with Mg in the present alloy, and Al11La3 has been found in other research after adding La to Mg-Al-Zn alloys [15, 18, 19]. Additionally, it is obvious that the peak of Al11La3 increases exponentially in the XRD patterns of the as-extruded AZ-0.3La alloys as Al increases in the blue frame, especially when the 2θ is around 35°.

3.2. Microstructural Observation of the As-Extruded Studied Alloys. Figure 3 displays the OM images and the magnified parts of as-extruded studied alloys, and the equiaxed grains could be observed in all the studied alloys, suggesting that the dynamic recrystallization occurs after the hot extrusion. According to the DRXed (dynamic recrystallized) grain size variation in Figures 3(b), 3(d), 3(f), and 3(h), the DRXed grain size decreases first and then remains unchanged as Al content increases, and range is about 9–15 μm. Besides, the DRXed grains are evenly distributed in the as-extruded AZ01-0.3La alloy, unevenly distributed in the as-extruded AZ31-0.3La and AZ61-0.3La alloys, and finally even distributed in the as-extruded AZ80-0.3La alloy. It could also be seen that all the precipitates are crashed and distributed along the extrusion direction in the as-extruded studied alloys. Some crushed precipitates with proper size and some segregated precipitates near the grain boundaries could be the potential nucleation sites and induce the recrystallization nucleation by the particle stimulated nucleation (PSN) mechanism during the extrusion process [22], as shown in Figures 3(b) and 3(d). Indeed, according to Figures 3(f) and 3(h), some new DRXed grains are observed in the as-extruded studied alloys with higher Al content (6% and 8%).
SEM images of the precipitates in the as-extruded studied alloys are shown in Figure 4, and the corresponding EDS analysis is displayed in Table 3, indicating that the crashed precipitates have the same crystal structure but different sizes compared to the as-cast studied alloys. Mg$_{12}$La particles are in the size of 1-2 $\mu m$, Al$_{11}$La$_3$ stubs are in the size of 2–5 $\mu m$, and Mg$_{17}$Al$_{12}$ are clustered into large particles with the size of 3–10 $\mu m$. Among them, the size of Al$_{11}$La$_3$ stubs increases as Al increases, which is about 1.5, 2.5, and 4 $\mu m$ in the as-extruded AZ31-0.3La, AZ61-0.3La, and AZ80-0.3La alloys, respectively. In addition, a small amount of Mg$_{12}$La particles in the submicron size are observed in the as-extruded AZ01-0.3La alloy.

Generally, the decrease of the DRXed grains is attributed to the impediment to the crystal boundary migration from the precipitates, which should be referred to as the broken Mg$_{12}$La particles, and the interaction of the Mg$_{17}$Al$_{12}$ phases and Al$_{11}$La$_3$ stubs in this study. In the as-extruded studied alloy, AZ01-0.3La alloy, only the broken Mg$_{12}$La particles exist in the Mg matrix, which could hinder the crystal

**Table 1:** Actual composition of the materials (wt. %).

| Alloys    | Zn   | Mn   | La   | Al   | Mg   |
|-----------|------|------|------|------|------|
| AZ01-0.3La| 0.548| 0.193| 0.289| 0    | Bal. |
| AZ31-0.3La| 0.935| 0.206| 0.309| 3.45 | Bal. |
| AZ61-0.3La| 0.840| 0.194| 0.279| 6.73 | Bal. |
| AZ80-0.3La| 0.601| 0.183| 0.273| 8.19 | Bal. |

Figure 1: Diagram of the technological process of the as-extruded studied alloy.
Figure 2: SEM images and XRD patterns of as-cast studied alloys: (a) AZ01-0.3La; (b) AZ31-0.3La; (c) AZ61-0.3La; and (d) AZ80-0.3La.
boundary migration and inhibit the dynamic recrystallization significantly compared with the as-extruded AZ01 alloy. In the as-extruded AZ31-0.3La, AZ61-0.3La, and AZ80-0.3La alloys, both of the broken Mg$_{17}$Al$_{12}$ phases and the Al$_{11}$La$_3$ stubs increase as Al increases, while the broken Mg$_{17}$Al$_{12}$ phases increase faster, leading to the decrease of the recrystallization temperature and the hinder to the crystal boundary migration, promoting the dynamic recrystallization nucleation, and reduce the grain sizes, respectively. In the as-extruded AZ61-0.3La alloy, although partial grains grew fast with large and sparse second particles, most grains were significantly refined with small and dense second particles, leading to the most refined grains.

### 3.3. Tensile Properties of As-Extruded Studied Alloys

Figure 5(a) shows the tensile properties of the as-extruded AZ-0.3La alloys at room temperature, and the corresponding properties of the as-extruded AZ alloys are also displayed in Figure 5(a) in order to analyze their applications. It could be seen that the $R_m$, $R_p$ of the as-extruded AZ-0.3La and AZ alloys almost show the same change trend except for the $R_p$ of the AZ01-0.3La. Among them, the increase of Al, the $R_m$ of both the as-extruded AZ-0.3La and AZ alloys increases, and the $A$ increases first and then decreases. It is noteworthy that the $R_p$ of the as-extruded AZ-0.3La alloys decreases first and then increases as Al increases, which is always increasing in the as-extruded AZ alloys. In addition, the strengths of the AZ01-0.3La and AZ31-0.3La alloys are significantly enhanced compared to the as-extruded AZ01 and AZ31 alloys, while the strengths of the as-extruded AZ61 and AZ80 alloys change little after adding 0.3% La, indicating that 0.3% La could enhance the DRXed grains according to the Hall–Petch relationship. Generally, the DRXed grains could be refined by adding RE elements attributed to the impediment to the crystal boundary migration from the precipitates, and the mechanical properties could be enhanced with the refined DRXed grains according to the Hall–Petch relationship. However, in this study, both of the broken Mg$_{17}$Al$_{12}$ phases and the Al$_{11}$La$_3$ stubs increased as Al increased in the as-extruded AZ-0.3La alloys, leading to the hindering of the DRXed grains and reducing the grain sizes. While the broken Mg$_{17}$Al$_{12}$ phases increase faster than the Al$_{11}$La$_3$ stubs, which decreases the recrystallization temperature and promotes the dynamic recrystallization nucleation. However, it should be noted that the formation of the Al$_{11}$La$_3$ could resume Al atoms and decrease Mg$_{17}$Al$_{12}$, resulting in the weakening of the solid solution strengthening effect of Al atoms and the refining effect of the broken Mg$_{17}$Al$_{12}$ phases. In the as-extruded AZ31-0.3La alloy, the refining effect of Al$_{11}$La$_3$ was more obvious than the weakened refining effect of broken Mg$_{17}$Al$_{12}$ phases, so that the DRXed grains are obviously more refined than the as-extruded AZ31 alloy. In the as-extruded AZ80-0.3La alloy, the weakened refining effect of broken Mg$_{17}$Al$_{12}$ phases by 0.3% La is negligible as many broken Mg$_{17}$Al$_{12}$ phases still existed, so that the 0.3% La could refine the DRXed grains while the refining effect is weaker, resulting in the unobvious decrease of the average grain sizes than the as-extruded AZ80 alloy. In the as-extruded AZ61-0.3La alloy, an equilibrium is achieved between the refining effect of Al$_{11}$La$_3$ stubs and the weakened refining effect of broken Mg$_{17}$Al$_{12}$ phases, so that the average grain sizes are almost the same as in the as-extruded AZ61 alloy. A similar phenomenon has been reported in another report [23].

Additionally, the grain sizes are refined in the as-extruded AZ-0.3La alloys with 0, 3%, and 8% Al, so their strengths should increase compared to the as-extruded AZ alloys. However, both the $R_p$ of the as-extruded AZ80-0.3La alloy change little, which is not consistent with the Hall–Petch relationship and may be attributed to the

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**Table 2: EDS analysis results of precipitates in as-cast studied alloys (in %).**

| Alloy        | Precipitate     | Mg | Al | La | Zn | Mn | Total |
|--------------|-----------------|----|----|----|----|----|-------|
| AZ01-0.3La   | A(Mg-La-Mn)     |    | 82.5|    | 17.1| 0.91| 9.42  |
|              | B(Mg$_{17}$La)  |    |    | 94.3| 5.08| 0.62|       |
| AZ31-0.3La   | C(Al$_{11}$La$_3$) | 22.8| 57.6| 15.1| 4.50|    | 100   |
| AZ61-0.3La   | D(Mg-Al-La-Mn)  | 50.7| 35.3| 10.9| 2.52| 0.58| 100   |
| AZ80-0.3La   | E(Al$_{11}$La$_3$) | 35.4| 47.8| 14.4| 2.40|    | 100   |
|              | F(Al$_{11}$La$_3$) | 56.6| 31.7| 10.6| 1.10|    | 100   |
|              | G(Mg-Al-La-Mn)  | 20.5| 52.0| 7.90|    | 19.6 | 100   |
insufficient and larger broken $\text{Al}_1\text{La}_3$ stubs impeding the dislocation movement poorly. The corresponding selected area diffraction (SAD) patterns are then displayed in Figure 6(b) to further confirm the above point. According to the representative EDS pattern in Figure 6(c), the broken stubs in the as-extruded AZ80-0.3La in Figure 6(a) are identified as $\text{Al}_1\text{La}_3$ ($a = 0.4431$ nm, $b = 1.013$ nm, and $c = 1.314$ nm), and no pile-up of dislocation is observed around the broken $\text{Al}_1\text{La}_3$ stubs in Figure 6(a). Indeed, as the Al increases, the larger broken $\text{Al}_1\text{La}_3$ stubs could make it easier to divide the Mg matrix, resulting in the descendant elongation growth of the as-extruded AZ-0.3La alloys. By the

Figure 3: OM images of as-extruded AZ-0.3La alloys: (a, b) AZ01-0.3La.; (c, d) AZ31-0.3La.; (e, f) AZ61-0.3La; and (g, h) AZ80-0.3La.
way, the significant enhancement of the strengths and elongation of the as-extruded AZ01-0.3La alloy could also be related to the Mg12La and submicron-sized Mg-La particles (see in Figure 4(b)), which has been reported in other similar researches [24, 25].

Besides the grain size and the morphology and size of phases, the texture influences the strength and elongation of wrought magnesium alloys as well. It has been reported that adding the RE element could weaken the texture of wrought magnesium alloys, resulting in an enlargement of the elongation but a decrease in the strength [26, 27]. Therefore, the texture evolution of the as-extruded alloys represented by 3% and 8% Al content was investigated, and the results are shown in Figure 7. The ED and TD are reported to the extrusion and transverse directions, respectively. It could be seen that the (0002) base texture is parallel to the ED and the (1010) prismatic texture is perpendicular to the ED in all the as-extruded alloys, while the intensity is somewhat different. With high Al content, the max intensity of the (0002) base texture in the as-extruded AZ80 and AZ80-0.3La alloys is 2.62 and 2.39, respectively, which is a little weaker with the 0.3%La addition, while there is no difference with the max intensity of the (1010) prismatic texture, which is separately 1.94 and 1.89. In addition, the (0002) base texture and (1010) prismatic texture distribution change a little before and after 0.3%La addition. That is to say, the (0002) base texture is weakened in the as-extruded AZ80-0.3La alloy, and the weakened texture might be another contributing factor to the lack of change of the strength. With low Al content, the max intensity of the (0002) base texture in the as-extruded AZ31 and AZ31-0.3La alloys is 2.38 and 2.60, respectively, which is a little stronger but more scattered after the 0.3%La addition, so it is doubtful that the (0002) base texture is strengthened. Besides, the max intensity of the (1010) prismatic texture is 1.92 and 1.61, respectively, indicating that the (1010) prismatic is weakened in the as-extruded AZ31-0.3La alloy.

In order to further analyze the texture evolution of the as-extruded AZ31 alloys after 0.3%La addition, the SF (Schmidt Factor) distribution statistics were studied, and the

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Table 3: EDS analysis results of precipitates in as-extruded AZ-0.3La alloys (in %).

| Alloy     | Precipitate | Mg   | Al   | La   | Zn | Mn | Total |
|-----------|-------------|------|------|------|----|----|-------|
| AZ01-0.3La| A(Mg-La-Mn) | 90.1 | —    | 8.31 | —  | 1.59| 100   |
| AZ31-0.3La| B(Al11La3)  | 42.8 | 44.3 | 12.7 | —  | 0.02| 100   |
| AZ61-0.3La| C(Al11La3)  | 78.8 | 16.8 | 4.40 | —  | —  | 100   |
| AZ80-0.3La| D(Al11La3)  | 30.9 | 51.9 | 14.4 | 2.80| —  | 100   |
| AZ31-0.3La| E(Al11La3)  | 12.0 | 66.1 | 18.4 | 3.50| —  | 100   |
| AZ61-0.3La| F(Mg17Al12) | 92.6 | 6.56 | 0.84 | —  | —  | 100   |
| AZ80-0.3La| G(Al11La3)  | 25.5 | 57.9 | 13.3 | 3.30| —  | 100   |

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Figure 4: SEM morphologies of as-extruded AZ-0.3La alloys: (a) AZ01-0.3La; (b) AZ31-0.3La; (c) AZ61-0.3La; and (d) AZ80-0.3La.
Figure 5: Tensile properties and the relationship between the average grain size and Al content of as-extruded AZ and AZ-0.3La alloys: (a) Property and (b) relationship.

Figure 6: TEM test of broken Al$_2$L$_a_3$ stubs in the as-extruded AZ80-0.3La alloy: (a) TEM image; (b) the corresponding SAD patterns; and (c) the EDS pattern.
Figure 7: Textures of the as-extruded AZ and AZ-0.3La alloys: (a) AZ31; (b) AZ31-0.3La; (c) AZ80; and (d) AZ80-0.3La.

Figure 8: Schmidt factor (SF) distributions of the as-extruded alloys: (a, c) AZ31 and (b, d) AZ31-0.3La.
results are displayed in Figure 8. It could be seen that there is little change in SF distribution in (10T0) prismatic texture, and the average SF value of the as-extruded AZ31 and AZ31-0.3La alloys is 0.438 and 0.453, respectively. However, there is a sincere difference in (0002) base texture, and the average SF value of the as-extruded AZ31 and AZ31-0.3La alloys is 0.179 and 0.365, respectively. In addition, the SF distribution is more uniform and the average SF value is higher in the as-extruded AZ31-0.3La alloy, indicating that the (0002) base slip is easier to start in the as-extruded AZ31-0.3La alloy, which could lead to better plastic deformation ability. The higher SF of the magnesium alloy improves the ductility along the ED via RE microaddition, as has been reported in other research [28–30].

4. Conclusion

The conclusion section clearly explains the main findings and implications of the work, highlighting its importance and relevance.

The precipitates, the dynamic recrystallization, the texture, and the mechanical properties at room temperature of the as-extruded AZ alloys (Al = 0, 3, 6, 8, wt.%) by 0.3% La addition was systematic and investigated in this work. The strengthening mechanism was discussed, and the major results were summarized. Mg12La with a lamellar structure precipitated in AZ01-0.3La alloy, while Al11La3 with a needlelike structure precipitated in AZ-0.3La alloy with 3%, 6%, and 8% Al. Both Mg12La and Al11La3 are crashed in the as-extruded studied alloys. A small amount of Mg31La particles are in the submicron size in the as-extruded AZ01-0.3La alloy, and the size of Al11La3 stabs increases as Al increases. The DRXed grain size decreases first and then remains unchanged as Al increases in the as-extruded AZ-0.3La alloys, which are refined compared to the as-extruded AZ alloys due to the impediment to the crystal boundary migration from the crashed Mg12La, Al11La3, and Mg17Al12 phases, except in the as-extruded AZ61-0.3La alloy. With Al increasing, the Rm of the as-extruded AZ-0.3La and AZ alloys increases at T90, and the Rm of the as-extruded AZ01-0.3La alloy is almost the same as the Rm at (205MPa) of the as-extruded AZ01 alloy. The A of the as-extruded AZ-0.3La alloys is greater than 20%, except for the as-extruded AZ01-0.3La alloy. With the refined grains and the weakened (0002) base texture and (10T0) prismatic texture, the as-extruded AZ31-0.3La alloy shows the best comprehensive tensile properties, of which the Rm is 162MPa, the Rm is 252MPa, and the A is 21.7%.

Data Availability

The experimental data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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