Microstructure and mechanical properties of Mg-2Zn-0.3Ca-0.5La magnesium alloy extruded with different temperatures

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Abstract
The extrusion tests of the Mg-2Zn-0.3Ca-0.5La (ZXLa200) alloy were carried out at different extrusion temperatures (260 °C–320 °C), and the mechanical properties and microstructure were studied and analyzed by means of optical microscope, scanning electron microscope, x-ray diffraction, transmission electron microscope, and tensile testing. The research have demonstrated significant influence of Extrusion temperature towards grain size, recrystallization grains and texture. With the extrusion temperature increases, the recrystallization grain of the extruded ZXLa200 alloy increases, but the texture intensity decreases. During the extrusion process, a large amount of Ca2Mg6Zn3 (50–200 nm) precipitates are precipitated, which inhibited the growth of dynamic recrystallization grains and improved the yield strength of the alloys. The tensile yield strength and elongation of extruded ZXLa200 alloy were acquired to be 375 MPa, 5.9% at 260 °C and 310 MPa, 19.2% at 320 °C.

1. Introduction
Magnesium is one of the most widely distributed elements in nature, ranking the 8th place (O, Si, Al, Fe, Ca, Na, K, Mg), accounting for about 2.35% of the crustal mass and is also the third most abundant dissolved mineral in seawater [1]. Due to its low density (1.74 g cm⁻³), high specific strength and excellent damping capacity, magnesium alloy has broad application prospects in automobile and aerospace industries [2]. However, the applications of magnesium alloys have been limited due to their higher cost or lower yield strength in comparison with some other alloys, such as Al alloys. The previous researches shows that the addition of RE, Ca, Zn, Si and other alloying elements can enhance the mechanical properties of Mg alloys due to the effects of solid solution-strengthening, dispersion-strengthening and precipitation-strengthening [3–5]. Among the multiple Magnesium alloy systems, Mg-Zn alloy is an ideal candidate for the base alloys to be microalloyed as low-cost, high-strength Mg alloy because of its abundance and excellent effect of aging strengthening and solution strengthening [6]. However, the pure Mg-Zn binary alloy has poor application value in practice and the third alloy element is usually selected to improve the processing performance and mechanical properties [7]. The addition of rare earth (RE) elements can effectively refine the microstructure, enhance the high temperature strength and creep resistance of the Mg-Zn alloy [8]. Du [9] pointed out that with the addition of La to the Mg-6Zn alloy, the Mg₂Zn₇ phase in the as-cast alloy is gradually replaced by Mg-Zn-La ternary phase of the orthogonal structure. Wu [10] suggested that adding Ce to ZK60 alloy can refine grains and purify grain boundaries. During the hot extrusion process, the recrystallized grain size has decreased with the increase of Ce content, the alloy structure is gradually refined, and the alloy’s tensile strength and elongation are improved. However, the elongation decreased when Ce content exceeds 1.0 wt.%. Ma [11] also found the similar phenomenon. Adding trace Ca into Mg-Zn alloy also can refine the grain and enhance heat resistance, reduce flammability and increase the strength [12–14]. Zhang [15] studied the structure and properties of Mg-1Zn-xCa (x=0, 0.2, 0.5wt.%) alloy after extrusion. The study found that adding a small amount of Ca stimulate nucleation and precipitation, refines the alloy grains and weakens the alloy...
texture, and the plasticity of the elongation of the extruded alloy can reach 38%. Comparing with single addition of trace elements, multiple additions are more effective for the modification of microstructure and improvement in mechanical properties of Mg-Zn alloys [9–18]. For example, the extruded Mg-6Zn-0.2Ca-0.8Zr alloy exhibits a finer microstructure and denser precipitate distribution than the extruded Mg-6Zn-0.2Ca ternary alloy [19]. Guo [16] investigated the effect of Ca additions on the microstructure and mechanical properties of as-extruded Mg-6Zn-3La alloy. It was found that comparing with the Ca-free alloy, the ultimate tensile strength, yield strength and elongation are improved by about 37%, 53% and 24% respectively.

The extrusion deformation process of magnesium alloy is also at the center of attention for current research field. Some scientific researchers have carried out further research work on magnesium alloy extrusion process. For instance, Bai [20] studied the influence of extrusion temperature on the microstructure and properties of magnesium alloys for automobiles. With the increase of extrusion temperature, the grains and larger fraction of recrystallized grains of the extruded Mg-6Zn-0.5Zr-xLa alloy increased, while the texture intensity and strength decreased according to Zengin’s research [21].

The extrusion temperature is one of the important parameters in the extrusion process and exerted significant effect on the microstructure and mechanical properties of deformed magnesium alloy. The potential slip system in magnesium alloy can be activated by elevation of temperature. But when the extrusion temperature is too high, not only the operation is inconvenient, but also the surface quality of the alloy is damaged by serious oxidation [22]. In addition, the deformation structure of magnesium alloy is very sensitive to temperature, but when the extrusion temperature is too low, the grains cannot be refined by dynamic recrystallization, and there are a large number of twins in the coarse grain structure. When the extrusion temperature is too high, secondary recrystallization may occur, resulting in grain growth.

In our previous study, Effects of Ca additions (0, 0.3 and 0.5 wt.%) on the microstructure and mechanical properties of the as-extruded Mg-6Zn-3La alloys were investigated by OM, XRD, SEM, TEM and tensile testing and found that fine Ca$_3$Mg$_6$Zn$_3$ particles can inhibit growth of the dynamic recrystallization grains during extrusion, which plays an important role on the improvement of yield strength [16]. The Mg-4Zn-0.5Ca-2RE alloy was studied by Naghdi [23], where the formation of Mg$_2$RE$_3$, Mg$_2$RE$_2$, and Ca$_3$Mg$_6$Zn$_3$ phases was reported. Du et al [24] also investigated the high-strength, low-cost wrought Mg-2.5 mass% Zn alloy through micro-alloying with Ca and La, where fine dynamically recrystallized grains and precipitates contributed to the enhancement of tensile strength. These excellent works indicate that the Mg-Zn-Ca–RE system is worthy of further study.

However, the effects of extrusion temperature on the properties and microstructure of Mg-Zn-Ca–RE series alloys were rarely studied. In order to obtain low-cost, high-strength magnesium alloy, it is necessary to explore an appropriate extrusion temperature parameter. In this paper, we choose Mg-2Zn alloy as the base alloy due to its excellent aging and solid solution strengthening effects. As mentioned above that the addition of rare earth (RE) elements, calcium [21, 22], and the combination of them [16–18, 23–30] have resulted in grain refinement, enhancement of mechanical properties, possibility of age hardening, and texture weakening. Amongst these RE elements, La are cheap and light RE elements with high potential to be employed for development of low-cost high-strength Mg alloys. Ca is also a cheap alkaline earth element due to the abundant reserve on earth. So, Ca and La which are cheap are choose as microalloying elements.

Therefore, an experimental Mg-2Zn-0.3Ca-0.5La (ZXLa200) alloy was subjected to extrusion at different temperatures, and the microstructure and mechanical properties were studied in this paper.

2. Experimental procedures

The alloy of Mg-2Zn-0.3Ca-0.5La (ZXLa200) was prepared by melting commercial pure Mg, Zn, Mg-25wt.% Ca and Mg-30wt.% La alloys in an electric resistance furnace under a mixed atmosphere of CO$_2$ and SF$_6$. After heat preservation at 760 °C for 40 min, the temperature was reduced to 730 °C, then poured the alloy liquid into the preheating mould at 250 °C to obtain the casting. The chemical composition of the as-cast alloy was determined by Mg-1.98Zn-0.32Ca-0.53La by the x-ray spectrometer. The ZXLa200 as-cast billets were homogenized at 350 °C for 20 h, followed by water quenching. The as-homogenized billets were extruded at 260, 280, 300 °C and 320 °C (named as ET260, ET280, ET300, ET320, respectively) to obtain the extruded rods, with the ram speed of 0.2 mm s$^{-1}$ and the extrusion ratio of 25. The extruded rods were cooled in air after exiting the die. The technological process of sample preparation is shown in figure 1.

The microstructure characteristics of the ZXLa200 as-cast, as-homogenized, as-extruded alloys were analyzed by optical microscope (OM, Olympus GX71), scanning electron microscope (SEM, Quanta 200 FEG), transmission electron microscope (TEM, JEM-3010) equipped with an energy dispersive spectrometer (EDS). The phase analyses were examined by x-ray diffraction (XRD, Rigaku D/max 2500PC) using a Cu Kα radiation. The incomplete (0002), (10–10), (10–11) and (10–12) pole figures were obtained by x-ray diffraction, and (0002) and (10–10) pole figures were calculated. The average method of DRX fractions and average DRX grain size of
the as-extruded rods was mentioned in the previous literature [16]. In OM and SEM observation, the solution of acid (5 ml water + 2.2 g picric acid + 5 ml acetic acid + 35 ml ethanol) was used.

The tensile properties of the extruded rods were performed by using the Instron 5569 universal test machine at a beam speed of 0.6 mm min$^{-1}$ (corresponding to the strain rate of $1 \times 10^{-3}$ s$^{-1}$) at the condition of room temperature. The diameter and the gauge length of these specimens was 2.5 mm and 25 mm, respectively, which was machined according to the testing standard of GB/T 16865-2013. The tensile tests of the sample corresponding to each temperature were repeated three times to ensure the accuracy of data. The microhardness (HV) was the average value of scattered 10 points (the maximum and minimum are removed from the total of 12 points) obtained by the microhardness tester (HVS-1000).

3. Results and discussion

3.1. Microstructure

In order to compare with the as-cast and as-homogenized alloys, the optical and SEM images of the as-cast alloy and as-homogenized alloy are provided in figure 2. The ZXLa200 alloy is composed of primary $\alpha$-Mg grains and the second phases distributing at grain boundaries as well as grain interior. The average grain size of the as-cast
alloy is approximately 90 μm from figures 2(a1) and (b1). Comparing with the as-cast ZXLa200 alloy, the grain size of the as-homogenized ZXLa200 alloy is about 110 μm with the second phases significantly reduced (shown in figures 2(a2) and (b2)), indicating the promotion of second phases partial dissolution due to homogenization treatment. It is certain that these secondary phase precipitates with a diameter larger than 1 micrometer can serve as nucleation sites for DRX grains in the process of extrusion based on a famous particle stimulating nucleation (PSN) mechanism [16, 25, 30].

Figure 3 has illustrated the optical micrographs and SEM images of the ZXLa200 alloys extruded at different temperatures (260 °C, 280 °C, 300 °C and 320 °C). Based on observation, the as-extruded alloys exhibited significant bimodal grain structure consisting of fine dynamic recrystallization (DRX) grain and rough...
non-dynamic recrystallization (non-DRX) region extension along the extrusion direction (ED). The detailed microstructural characteristics of the as-extruded alloys at different temperatures are shown in table 2. It can be seen that the extrusion greatly influences the DRX fraction and grain size. Specifically, the average size \( d_{\text{DRX}} \) and area fraction \( f_{\text{DRX}} \) of recrystallized grains of ET260 alloy are estimated to be \( \sim 0.48 \) \( \mu \text{m} \) and \( \sim 58.3\% \).

The \( d_{\text{DRX}} \) and \( f_{\text{DRX}} \) of recrystallized grains increase gradually with the increase of extrusion temperature. When the temperature reaches 320 °C, the \( d_{\text{DRX}} \) and \( f_{\text{DRX}} \) has reached \( \sim 1.21 \) \( \mu \text{m} \) and \( \sim 81.2\% \). In figure 3(b), there were some unevenly distributed precipitates, which were produced by hot extrusion. Comparing with ET300 or ET320, there are less precipitates in ET260 because of its slower precipitation kinetics at the lower extrusion temperature [25]. In fact, grain growth and dynamic recrystallization during extrusion are both thermally activated processes, so the driving force for grain growth and dynamic recrystallization increases with the increment of billet temperature. In this case, the distributed precipitates pinning force on the grain boundary is also weakened. Combining with these reasons, the final grain size and \( f_{\text{DRX}} \) with the increase of extrusion temperature.

Figure 4 shows the x-ray diffraction patterns of the ZXLa200 alloys in the as-cast, as-homogenized and as-extruded conditions. It can be observed that there are peaks of MgZn2 phase and \( \text{Ca}_3\text{Mg}_6\text{Zn}_3 \) phase in the extruded alloy, but the strength of peaks is very feeble due to the small content of these phases. Besides, there is a distinct unknown phase of Mg-Zn-La-Ca in the alloy. Similar phase was also found in the study conducted by Guo [16].

EM/EDS analysis is carried out for the as-cast, as-homogenized and as-extruded alloys, and the results are provided in figure 5 and table 1. Figure 5 illustrates SEM/EDS analysis of the as-cast, as-homogenized and as-extruded ZXLa200 alloys. For the as-cast ZXLa200 alloy (figure 5(a)), the second phases are distributed at the triple junction (Point A) and inside the grain (Point C), containing Mg, Zn, La and Ca. The unknown phase is most likely to be (Mg, Zn, RE, Ca) phase [16]. Other researchers [26] also discovered the similar situation in

![Figure 4. The x-ray diffraction patterns of the ZXLa200 alloys.](image)

| Table 1. EDS analysis of the ZXLa200 alloys. |
|--------------------------------------------|
| Element (at.%) |
| Position     | Mg  | Zn  | La  | Ca  |
|---------------|-----|-----|-----|-----|
| A(figure 4(a))| 82.91 | 10.98 | 3.16 | 2.94 |
| B(figure 4(a))| 85.75 | 8.51 | —   | 5.74 |
| C(figure 4(a))| 87.66 | 8.11 | 2.81 | 1.42 |
| D(figure 4(b))| 87.54 | 9.23 | 2.75 | 0.47 |
| E(figure 4(b))| 90.11 | 7.11 | 1.55 | 1.23 |
| F(figure 4(b))| 72.81 | 20.09 | 6.23 | 0.77 |
| G(figure 4(c))| 86.36 | 10.26 | 3.16 | 0.22 |
| H(figure 4(c))| 95.99 | 2.86 | —   | 1.15 |
| I(figure 4(c))| 97.46 | 1.89 | 0.45 | 0.20 |
| J(figure 4(c))| 97.38 | 1.92 | 0.53 | 0.17 |
Mg-Zn-Ca-RE alloy. But La is not detected in the second phase at Point B, combined with XRD in figure 4, it can be confirmed as Ca$_2$Mg$_6$Zn$_3$, which is in accordance with the previous researches [19, 24, 31] indicating that there are at least two types of second phase in the ZXLa200 alloy. The phases (Point D, E and F) are similar to points A and C, but the Ca$_2$Mg$_6$Zn$_3$ phase is not detected in the as-homogenized ZXLa200 alloy (figure 5(b)), which implies that the excess Ca$_2$Mg$_6$Zn$_3$ phase may dissolve in the matrix after solid solution treatment. For the as-extruded ZXLa200 alloy (figure 5(c)), identical phenomenon can be also found at the points G, H, I and J, which further confirmed that Mg-Zn-La-Ca and Ca$_2$Mg$_6$Zn$_3$ phase are present in ZXLa200 alloy. The Ca$_2$Mg$_6$Zn$_3$ phase is detected in the as-extruded ZXLa200 alloy, which may be due to the dynamic precipitation of the second phase during extrusion.

For further analysis of the precipitates, the TEM micrographs of the ET320 alloy were shown in figure 6. There are many dispersed secondary phase particles distributed either in grain interiors or at grain boundaries uniformly in the DRX region (figure 6(a)). These numerous secondary phase particles can effectively block the dislocations movement and pin the grain boundaries during plastic deformation, thereby increasing the yield strength of the alloy. The low-magnification bright field image (figure 6(b)) shows a small amount of the micron coarse blocky precipitation with the length of these coarse precipitates approximately 2 um. According to the literature [7, 16, 28] and the results of the EDS (figure 6(c)), the micron-scale second phase particles (point A in figure 6(b)) are determined as unknown Mg-Zn-La-Ca phase. The figure 6(d) shows the fine second phase particles in the nanometer dimension (50–200 nm) and exhibiting a relatively uniform distribution in the α-Mg matrix. As can be seen from the figure, there are two distinct secondary phases. The point B is determined as Ca$_2$Mg$_6$Zn$_3$ phase by micro-diffraction and EDS (figure 6(e)). According to the high-resolution transmission electron microscope (HRTEM), the diffraction pattern obtained by fast Fourier transform (FFT) and the corresponding XRD result, this representative nano-scale particle (point C) is identified as the MgZn$_2$ phase. Guo et al [16] also reported Ca$_2$Mg$_6$Zn$_3$ and a spot of MgZn$_2$ precipitates in the extruded Mg-6Zn-3La-0.5Ca alloy.

Figure 7 shows the experimental pole figures of the as-extruded alloys. The as-extruded ZXLa200 alloys present rod textures with the basal planes parallel to the extrusion direction, which is a classical circumstances of the extruded Mg alloy [17]. The maximum texture intensities gradually decreases with the increase of extrusion temperature. In particular, a maximum texture intensity of 11.247 is obtained in the ET260 alloy. While the maximum texture intensity of ET300 and ET320 alloys are only 8.821 and 7.358, which reduce by 10.5% and 34.6% respectively compared to ET260 alloy. It is widely acknowledged that in extruded magnesium alloys, DRX
Figure 6. TEM micrographs of the as-extruded ET320 alloy: (a) HAADF image of the DRX region, (b) bright-field image, (c) the EDS of the second phases arrowed as A in (b), (d) enlarged bright-field image, (e) microdiffraction of the fine precipitate arrowed as B in (d), and (f) the EDS of the fine precipitate arrowed as B in (d), (g) the HRTEM of fine precipitate arrowed as C in (d).

Figure 7. Experimental pole figures of the as-extruded ZXLa200 rods: (a) ET260, (b) ET280, (c) ET300, (d) ET320.
grains generally have weaker texture than non-DRX grains due to the more randomized DRX grains [30]. In other words, the weakening texture is related to the increase of DRX area fraction with the increase of the extrusion temperature (figure 3(a)). On the other hand, the dynamic precipitates during hot extrusion provided more random participates orientations, thus causes changes and randomization of the overall texture. Compared with ET300 or ET320, there are fewer precipitates in ET260 because of its slower precipitation kinetics at the lower extrusion temperature, so the texture intensity was stronger.

In general, more uneven deformation zones appear near the coarse particles during deformation, and these zones have large differences in microscopic orientation, which promotes nucleation of recrystallization, known as particle stimulated nucleation (PSN) [16, 25, 30]. As can be seen from figure 2, the ZXLa200 alloy all have a large number of residual coarse secondary phase after homogenization and these secondary phase can promote the nucleation of recrystallization by particle stimulated nucleation during extrusion. Fair amount of fine Ca2Mg6Zn3 particles are distributed along grain boundaries in the ZXLa200 alloy during extrusion (figure 6(a)) and effectively restrained the grain growth of dynamic recrystallization. The interaction between PSN effect of coarse unknown Mg-Zn-La-Ca phases and inhibition effect of Ca2Mg6Zn3 on grain growth leads to significant grain refinement in the extruded ZXLa200 alloy.

### 3.2. Mechanical properties

Figure 8 shows the tensile stress-strain curves of the four as-extruded ZXLa200 alloys and the corresponding mechanical property values are presented in table 3. ET260 exhibits the highest strength and microhardness, and
the values of YS, UTS and HV are 375, 384 MPa, and 86.6, respectively. As shown in figure, the strength decreases with extrusion temperature increment, but the ductility is significantly improved. Especially when the extrusion temperature is from 300 °C to 320 °C, the TYS of the alloy does not decrease much (from 325 to 310 MPa), while the elongation is significantly improved (from 14.6% to 19.2%). ET320 has the best elongation result of 19.2%, which is more than three times of ET260 (5.9%).

The high tensile strength of ET260 alloy extruded can be majorly explained by several strengthening mechanisms such as grain refinement strengthening, texture strengthening and precipitation strengthening.

As the grain size decreases, the number of grain boundaries will increase as well, which will bring greater adversity to the movement of dislocations, eventually leading to an increase in yield strength. The ET260 alloy has the finest DRX grain (∼0.48 μm) among all researched alloys. Therefore, for ET260 alloy, the contribution of fine-grain strengthening to mechanical strength is the largest. Moreover, the k value in equation (1) is also closely related to the texture intensity. The strong texture intensity own higher k value than those with a weak one in Mg alloy by Yu [32] reported. It should be seen from figure 7 that the texture intensity decrease with the increase of extrusion temperature. Therefore, the ET260 alloy with the finest grains and the highest texture intensity exhibits the most significant improvement in strength by the fine grain strengthening mechanism.

The precipitation strengthening is also a factor that greatly affects the yield strength. The Mg-2Zn-0.3Ca-0.5La alloy contains a large number of fine precipitates (mainly Ca2Mg6Zn3 phase), which were evenly distributed at grain boundaries and in grain interiors. These precipitates can increase yield strength via blocking the dislocation movement and causing dislocation pile-ups during plastic deformation.

In addition, texture also plays a prominent role on yield strength of wrought Mg alloys. Through unidirectional deformation, grain growth or recrystallization (dynamic and/or static), these alloys usually form grains with a preferred crystallographic orientation [27]. Such grains with a specific orientation will also increase the activation stress for the basal slip under tension along ED and induce higher strength of the alloy. With the temperature of extrusion increases from 260 °C to 320 °C, the texture intensity continues to decline, so ET260 alloy has the most significant texture hardening effect.

Figure 9. SEM fracture micrographs of the as-extruded ZXLa200 alloys: (a) ET260, (b) ET280, (c) ET300, (d) ET320.
The tensile elongation of the as-extruded ZXLa200 alloys increased from 5.9% to 19.2% with the extrusion temperature increases from 260 °C to 320 °C. It has been noticed that the significant improvement in elongation depends on the degree of dynamic recrystallization. As exhibited in figure 3, the area fraction of recrystallized grains of the as-extruded ZXLa200 alloys can be improved and texture intensity decreased with increment of extrusion temperature. In randomized grains, both basal and non-basal slip systems are more beneficial to be activated [53]. Therefore, the randomization of texture is of great significance for optimizing the ductility of the Mg alloy.

The SEM fracture micrographs of the as-extruded Mg-2Zn-0.3Ca-0.5La alloys are shown in figure 9. The tensile fracture surfaces of all alloys are rough and contains a large number of dimples, which is the typical ductile fracture characteristics [16]. As the extrusion temperature increases, the small dimples on the fracture surface of the alloys become deeper and the big dimples become denser. It can be found from the figure that some particles of second phase have produced microcracks. This is due to the deformation between the second phase particles and the matrix does not match during the tensile deformation process, microcracks will initiate on the interface firstly. Then they will further aggregate and grow up rapidly, eventually leading to fracture failure of the alloy.

4. Conclusions

The study investigates the microstructure and mechanical properties of the ZXLa200 alloy extruded at different temperature. The main conclusions are summarized as follows:

1. After solution treatment, the grain size of ZXLa200 alloy grows slightly and the number of second phase is significantly reduced.

2. The ZXLa200 alloy extruded at ET260 shows ultrafine DRX grains (~0.48 μm). With the increment of the extrusion temperature, the area fraction of recrystallized grain have improved while the DRX grains and texture intensity have decreased. The ZXLa200 alloys precipitate abundant fine Ca2Mg6Zn3 precipitates, with the precipitate size of 50–150 nm, are evenly distributed at grain boundaries and in grain interiors. These precipitates can increase yield strength via blocking the dislocation movement and causing dislocation pile-ups during plastic deformation.

3. ET260 exhibits the highest TYS, highest UTS and fair ductility of 375, 384 MPa and 5.9%, respectively. With the increase of extrusion temperature, the strength of the extruded ZXLa200 alloy decreased, while in contrast, the ductility has increased. ET320 has the best elongation performance of 19.2% and TYS has 310 MPa.

4. The strength enhancement of the extruded ZXLa200 alloy is mainly attributed to three factors: grain refinement strengthening, texture strengthening and precipitation strengthening.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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