Effects of Zn Addition on the Microstructure and Mechanical Properties of As-Extruded Mg-2Al-0.5Ca Alloy

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Abstract: The effects of Zn addition on the microstructure and tensile properties of as-extruded Mg-2Al-0.5Ca-xZn (x = 0, 0.3, 0.6, 0.9 wt.%) alloys were investigated in this work. The results showed that the extruded sheets exhibited a completely dynamically recrystallized microstructure, the grain size was refined, and texture weakening was achieved with Zn addition because of the segregation of Zn atoms on grain boundaries, which suppresses the growth of dynamic recrystallized grains. The addition of 0.6 wt.% Zn improved both the tensile strength and ductility of the as-extruded Mg-2Al-0.5Ca alloy. The as-extruded Mg-2Al-0.5Ca-0.6Zn alloy showed a 0.2% proof stress of 145 MPa, an ultimate tensile strength of 317 MPa, and an elongation of 30.0% along the extruded direction. The simultaneous improvement of strength and ductility was mainly due to the fine and homogeneous grain microstructure and the weakened extrude direction (ED)-tilted texture. The as-extruded Mg-2Al-0.5Ca-0.6Zn alloy showed little in-plane anisotropic tensile properties, with a 0.2% proof stress, ultimate tensile strength, and elongation in the 45° direction of 148 MPa, 299 MPa, and 25.0%, and those in the transverse direction of 148 MPa, 269 MPa, and 16.8%, respectively.

Keywords: Mg-2Al-0.5Ca alloy; extrusion; microstructure; dynamic recrystallization; texture

1. Introduction

Magnesium (Mg) alloy is the lightest commercial structural material for engineering applications such as transportation, aerospace, and 3C (consumer electronics, computer, and communication) industries [1–5]. Conventional wrought alloy, e.g., AZ31, usually forms a strong basal texture with the (0002) basal poles parallel to the normal direction (ND) after the hot extrusion or rolling process. This texture characterization limits the secondary forming processes such as sheet pressing. Therefore, modifying the texture features and improving the mechanical properties of Mg alloys have attracted many researchers to the Mg alloys research field over the past decades.

Alloying is a useful method to modify Mg alloys’ texture, and it is widely reported that Mg alloys containing rare earth (RE) elements show a weak basal texture because of the RE elements’ solute segregation and drag effect on grain boundaries [6–10]. Although the addition of RE elements could improve the mechanical properties of the Mg alloys, the high cost of REs is the limitation for its wide application. Ca is an inexpensive alloying element compared to RE elements, and it has been reported that Mg alloys with Ca can exhibit...
an ED-split texture component similar to RE-containing alloys after the hot deformation process [11–13]. In the past decade, many researchers have investigated the microstructures and mechanical properties of the wrought Mg-Al-Ca-based alloys [14–18]. Sandlubes et al. developed a rolled Mg-1Al-0.1Ca (wt.%) alloy sheet, which showed superior mechanical properties with an ultimate tensile strength of ~220MPa and elongation of about 20% [19]. The study showed that Al and Ca additions alter the deformation mechanism, activate the pyramidal slip activity, in addition to the basal <a> slip. Kim et al. revealed that 0.5 wt.% Ca addition in AZ31 alloy can weaken the basal texture and enhance the activity of nonbasal slip systems, contributing to the enhanced mechanical properties [20]. Ding et al. also revealed that the Ca element can weaken the texture and improve the ductility of rolled Mg alloy sheets [21]. Kamado et al. developed a wrought Mg-1.3Al-0.3Ca-0.4Mn (wt.%) alloy, which showed an ultimate tensile strength of 306 MPa and an elongation of 20% after age-hardening [14]. Concerning the low cost and low-alloyed development of Mg alloys, we calculated the composition of the Mg-2Al-0.5Ca-based alloys to avoid the Mg_{17}Al_{12} precipitates while retaining the Al_{2}Ca phase. Furthermore, Zn is also a promising element to influence the texture evolution and mechanical properties of Mg alloys [22–25]. Zhao et al. [26] reported that the intensity of the basal texture weakened as the Zn content increased from 1 to 4 wt.% in the as-extruded Mg-xZn alloys. Nakata et al. [27] discovered that adding 1 wt.% Zn into the Mg-8Al-1Ca-0.3Mn alloy can refine the grain size and improve its mechanical properties. However, in the Mg-Al-Zn-based alloys, when a Zn content over 1 wt.% would make the weldability of the Mg alloy worse [28], the Zn content should be limited to within 1 wt.%. As we know, extrusion is a common thermo-mechanical process to improve the performance of Mg alloys [29–32]. Based on the aforementioned information, in this work, we developed a novel extruded Mg-2Al-0.5Ca (AX21) alloy with different Zn contents (0, 0.3, 0.6, and 0.9 wt.%) to obtain comprehensive performance with high ductility and strength. The purpose was to reveal the influence of Zn addition on the microstructure evolution and tensile properties of the as-extruded Mg-2Al-0.5Ca alloy, and hopefully provide a guide to develop new low-cost wrought Mg alloys with medium strength and high ductility. Compared to commercial AZ31 alloy, the Mg-2Al-0.5Ca-based alloy has a lower content of alloying additions and low-density Ca. It will have more advantages in applications if the Mg-2Al-0.5Ca-based alloy has superior mechanical properties.

2. Materials and Experimental Procedure

Four experimental Mg-2Al-0.5Ca-xZn (x = 0, 0.3, 0.6, 0.9, all in wt.%) ingots were prepared from high-purity Mg (99.95 wt.%), pure Al (99.9%), pure Zn (99.99 wt.%), and Mg-20 wt.% Ca master alloys. The raw materials were melted in a steel crucible under a CO_{2}+1vol.%SF_{6} atmosphere at 720 °C, and the melts were maintained at ~720 °C for 15 min and then cast into a preheated cylindrical mold at 250 °C. The actual chemical compositions of the experimental alloys were measured by X-ray fluorescence spectrometry (XRF-1800 Shimadzu, Japan), and the results are shown in Table 1. The cast ingots were homogenized at 400 °C for 24 h and water-quenched. Afterward, the ingots were extruded at 300 °C with a ram speed of 1 mm/s and an extrusion ratio is 32. Consequently, sheets 56 mm in width and 5 mm in thickness were obtained.

| Nominal Composition | Measured Composition (wt.%) |
|---------------------|-----------------------------|
|                     | Al  | Ca  | Zn  | Mg  |
| Alloy 1: Mg-2Al-0.5Ca | 2.29| 0.64| -   | Bal.|
| Alloy 2: Mg-2Al-0.5Ca-0.3Zn | 2.21| 0.51| 0.28| Bal.|
| Alloy 3: Mg-2Al-0.5Ca-0.6Zn | 2.35| 0.49| 0.56| Bal.|
| Alloy 4: Mg-2Al-0.5Ca-0.9Zn | 2.22| 0.53| 0.91| Bal.|

Table 1. The nominal compositions of the experimental alloys.
The tensile tests were carried out on a uniaxial test machine (CMT6305-300 kN, SUNSTEST, Shenzhen, China) with a cross-head speed of 1.5 mm/min (strain rate is 0.002 s⁻¹) at room temperature (RT) according to ASTM standard E8-2004. Tensile samples had a gauge length of 12 mm and cross-sectional areas of 6 mm × 5 mm. To ensure reproducibility, three tensile samples were repeated, and the results were presented as the average.

Microstructure observation was performed using an optical microscope (OM, ZEISS Axiovert 40 MAT, Jena, Germany) and a scanning electron microscope (SEM, JEOL JSM-7800F, Tokyo, Japan). For the electron backscatter diffraction (EBSD) observation, the samples were mechanically ground and polished in the AC2 solution at −25 °C with a potential of 20 V for 90 s. Phase identification was performed on a D/Max2500 X-ray diffraction instrument (Rigaku, Toyko, Japan) at 36 kV, and the scan speed was 4°/min. The transmission electron microscopy (TEM) observations were performed on a Talos F200X G2 microscope (Thermo Fisher Scientific Inc., Waltham, MA, USA) at an accelerating voltage of 200 kV.

3. Results

3.1. Microstructures of As-Cast Alloy

Figure 1 shows the SEM images of the as-cast samples of Mg-2Al-0.5Ca-xZn (x = 0, 0.3, 0.6, 0.9 wt.%) alloys. It can be seen that the second phases are distributed in all the alloy samples. The second phases, which are pointed out by cross marks A-D in Figure 1, were examined by EDS, and the corresponding EDS analysis results are presented in Table 2, which indicates that the stoichiometry of the second phase is near Al₂Ca. Figure 2 shows the X-ray diffraction spectra of the as-cast alloys, and it illustrates that the as-cast alloys are composed of α-Mg and Al₂Ca phases. The Mg-2Al-xCa (x, 0–1 wt.%) phase diagram is shown in Figure 3, which was calculated by Pandat software (CompuTherm, Middleton, WI, USA). With the fixed Al content of 2 wt.% and varied Ca (0–1 wt.%) content, it was found that Al₂Ca phases are the main precipitates in the Mg-2Al-0.5Ca alloy.

3.2. Microstructures of the As-Extruded Alloy

The OM images of the as-extruded sheet samples in the ED–TD plane are shown in Figure 4, where ED is the extrusion direction and TD is the transverse direction. It can be seen that all the as-extruded sheets show complete equiaxed DRXed grains. The average grain size of as-extruded Zn-contained alloys is refined compared to Zn-free alloys. In addition, many second phases are observed in the SEM images (seen in Figure 5), where these second phases distribute along the extrusion direction of the as-extruded samples. According to the EDS results (Table 3) and XRD analysis (Figure 6), these second phases are Al₂Ca, which are the intermetallic phases and are broken up into particles during the extrusion process.
Figure 1. SEM images of the as-cast alloys: (a) alloy 1, (b) alloy 2, (c) alloy 3, and (d) alloy 4. A–D are point EDS of the second phases.

Table 2. EDS analysis of the second phases in as-cast Mg-2Al-0.5Ca-xZn alloys (at %).

| Position | Mg    | Ca   | Al   | Possible Phases |
|----------|-------|------|------|-----------------|
| A        | 76.12 | 7.72 | 16.16| Al₂Ca           |
| B        | 66.53 | 9.55 | 23.92| Al₂Ca           |
| C        | 70.03 | 9.58 | 20.38| Al₂Ca           |
| D        | 49.55 | 15.49| 34.96| Al₂Ca           |

Figure 2. X-ray diffraction spectra of the as-cast alloys.
3.2. Microstructures of the As-Extruded Alloy

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Figure 5. SEM micrographs of the extruded sheets: (a) alloy 1, (b) alloy 2, (c) alloy 3, and (d) alloy 4. A–D are point EDS of the second phases.

Table 3. EDS analysis of the second phases on the extruded sheets marked in Figure 4.

| Position | Mg   | Ca   | Al   | Possible Phases |
|----------|------|------|------|-----------------|
| A        | 75.5 | 8.29 | 16.21| Al₂Ca           |
| B        | 41.7 | 16.58| 41.72| Al₂Ca           |
| C        | 61.33| 9.7  | 28.96| Al₂Ca           |
| D        | 50.74| 12.86| 36.4 | Al₂Ca           |
To more accurately illustrate the microstructure of the as-extruded sample, EBSD inverse pole figure (IPF) maps, (0002) basal pole figures, and grain size distribution maps of the as-extruded Mg-2Al-0.5Ca-xZn samples are shown in Figure 7. It can be seen that all the samples display fully recrystallized microstructures, and the average grain size of alloy 1, alloy 2, alloy 3, and alloy 4 is 8.8 µm, 6.1 µm, 4.3 µm, and 5.7 µm, respectively. According to the (0002) basal pole figure, it can be seen that the (0002) basal poles are tilted ~±20° from the ND toward the ED. In addition, when the Zn addition increases, the (0002) basal poles spread toward the TD slightly. In addition, the maximum texture intensity of alloy 1 is ~13.7 mrd, and texture intensities decrease to 8.4~8.9 after Zn addition. It is widely reported that the segregation of solute atoms along the grain boundary (GB) could suppress the recrystallization rate, and result in a weak texture [33,34]. Figure 8 shows the HAADF-STEM image and corresponding EDX maps of the as-extruded Mg-2Al-0.5Ca-0.6Zn alloy. It shows the obvious segregation of Zn atoms along GBs, but the Ca segregation is not observed. It is hypothesized that Al2Ca particles, which are formed during the solidification, consume the majority of Ca elements, and the Al2Ca particles are very stable because it has a high melting point (1079 °C). These Al2Ca particles will not decompose or transform to other phases during the homogenization and extrusion process; thus, there are few Ca elements in the Mg matrix. The segregation of Zn atoms along GBs reduces the grain boundary mobility and enhances the solute dragging effect [35], thus suppressing the growth of dynamic recrystallized (DRX) grains and weakening the recrystallization texture [35–37]. It is widely reported that texture weakening is conducive to a high ductility of Mg alloy [38–41]. In this study, 0.6 wt.% Zn addition in the Mg-2Al-0.5Ca alloy not only weakens the texture but also refines the grain size, which are both beneficial to the improvement of the ductility of the Mg-2Al-0.5Ca alloy. Therefore, we can conclude that Zn addition could refine the grain size and weaken the texture intensity of the as-extruded Mg-2Al-0.5Ca alloy.

Figure 6. X-ray diffraction spectra of the extruded sheets.
Figure 7. Inverse pole figure, pole figure, and grain size distribution maps of the sheets: (a) alloy 1, (b) alloy 2, (c) alloy 3, and (d) alloy 4.
3.3. Mechanical Properties

Figure 9 shows typical true stress–strain curves of the as-extruded sheets stretched along the ED, 45° direction, and TD, respectively. The values of 0.2% yield strength (YS), ultimate tensile strength (UTS), and elongation to failure (ε) are presented in Table 4. As shown in Figure 7a, alloy 1 exhibits a YS and UTS of 110 and 236 MPa along the ED, respectively, and the YS and UTS are 139 and 271 MPa for the 45° direction and 119 and 248 MPa for the TD, respectively. The ε along the ED, 45° direction, and TD are 20.1%, 21.7%, and 16.1%, respectively. With the addition of 0.3 wt.% Zn, alloy 2 shows obvious improvements in YS and UTS along the ED, which are 142 and 297 MPa, respectively, and the ε is 23.3%; these are larger than those of alloy 1. As shown in Figure 7c, alloy 3 shows a similar YS for all three loading directions; the YS, UTS, and ε of alloy 3 are simultaneously enhanced to 145 MPa, 317 MPa, and 30.0% along the ED; 148 MPa, 299 MPa, and 25.0% along the 45° direction; and 148 MPa, 269 MPa, and 16.8% along the TD, respectively. As shown in Figure 7d, when the Zn addition is 0.9 wt.%, alloy 4 shows a YS, UTS, and ε of 149 MPa, 327 MPa, and 28.6% along the ED and 152 MPa, 258 MPa, and 13.6% along the TD, respectively. By comparing YS vs. ε for alloy 3 and alloy 4, although alloy 4 shows...
a little higher strength compared to alloy 3, the ductility of alloy 4 is lower than that of alloy 3. As observed, dilute addition of Zn could improve the strength and ductility of the as-extruded sheet, indicating that Zn micro-alloying is an effective method to improve the mechanical properties of Mg-Al-Ca-based alloys. When the Zn addition is 0.6 wt.%, the alloy shows a good balance between strength and ductility.

![Figure 9](image1)

**Figure 9.** Typical true stress–strain curves of as-extruded sheets: (a) alloy 1, (b) alloy 2, (c) alloy 3, and (d) alloy 4.

| Alloy  | ED | 45° | TD |
|-------|----|-----|----|
|       | $\sigma_{0.2}$ | $\sigma_b$ | $\varepsilon$ | $\sigma_{0.2}$ | $\sigma_b$ | $\varepsilon$ | $\sigma_{0.2}$ | $\sigma_b$ | $\varepsilon$ |
| Alloy 1 | 110 ± 3 | 236 ± 5 | 20.1 ± 0.8 | 139 ± 2 | 271 ± 3 | 21.7 ± 0.6 | 119 ± 2 | 248 ± 2 | 16.1 ± 0.4 |
| Alloy 2 | 142 ± 2 | 297 ± 4 | 23.3 ± 0.6 | 140 ± 2 | 285 ± 4 | 23.1 ± 0.5 | 142 ± 3 | 245 ± 2 | 13.9 ± 0.8 |
| Alloy 3 | 145 ± 2 | 317 ± 4 | 30.0 ± 0.4 | 148 ± 2 | 299 ± 3 | 25.0 ± 0.3 | 148 ± 2 | 269 ± 4 | 16.8 ± 0.6 |
| Alloy 4 | 149 ± 2 | 327 ± 3 | 28.6 ± 0.5 | 142 ± 2 | 297 ± 2 | 24.1 ± 0.4 | 152 ± 2 | 258 ± 3 | 13.6 ± 0.5 |

$\sigma_{0.2}$: MPa; $\sigma_b$: MPa.

Table 4. Tensile properties of the alloys at room temperature (RM).

Figure 10 shows the tensile fracture surfaces of as-extruded sheet samples stretched along the ED. It can be seen that there are many shear cleavage planes and tear ridges on the fracture surface of alloy 1, which shows a quasi-cleavage transgranular fracture. With the
addition of 0.3 wt.% Zn, some dimples appear on the fracture surface of alloy 2. With the Zn content reaching 0.6 wt.%, the fracture surfaces of alloy 3 contain numerous fine-scale deep dimples, which means that alloy 3 shows a ductile fracture. With the further increase in Zn content, the dimples on the fracture surface of alloy 4 decrease, which means the elongation of alloy 4 decreases again. As a result, it can be concluded that the trace Zn addition causes significant changes in the tensile fracture surface of the as-extruded Mg-Al-Ca alloys, and these changes are consistent with the performance of the alloys’ ductility.

Figure 10. Tensile fracture surfaces of as-extruded alloy sheets stretched along the ED: (a) alloy 1, (b) alloy 2, (c) alloy 3, and (d) alloy 4.

4. Discussion

It is widely reported that grain size reduction, solid solution strengthening, precipitation strengthening, and second-phase strengthening could increase the strength of the Mg-based alloys [31,42]. In this work, Mg-2Al-0.5Ca alloys with Zn addition showed better mechanical properties compared to the Zn-free alloy. According to the microstructural analysis above, grain boundary strengthening and solid-solution strengthening were two of the dominant factors responsible for the enhanced YS of the Mg alloys.

Figure 11 shows the effect of grain size on the yield stress of the extruded alloys. It can be seen that the smaller the grain size, the higher the strength of the alloy. According to the Hall–Petch equation \( \Delta \sigma_{gy} = kd^{-1/2} \) [43], where \( k \) is the Hall–Petch constant and \( d \) is the average grain size, we can calculate the Hall–Petch slope as 254 MPa/\( \mu m^{0.5} \). This value is consistent with the reported values for Mg alloys [43]. Thus, the grain boundary strengthening (\( \Delta \sigma_{gy} \)) can be calculated to be 85.6 MPa, 102.9 MPa, 122.5 MPa, and 106.4 MPa for
extruded alloy 1, alloy 2, alloy 3, and alloy 4, respectively. Therefore, when the Zn content is 0.6 wt.%, the grain size of alloy 3 is smallest, and the grain boundary strengthening is increased by 37 MPa.

![Figure 11](image-url)

**Figure 11.** The corresponding Hall–Petch plot.

The solid solution strengthening effect of solutes arises from the size misfit and modulus misfit parameters. The Zn atom is recognized to be one of the effective solute atoms for increasing the strength when it is added into Mg alloys [44], because of the difference in the atomic radius between Zn and Mg, and the wide range of solubility. Regardless of whether the Mg alloy undergoes plastic deformation or not, its intrinsic crystal structure does not change. Namely, the lattice distortion and modulus effect caused by solution atoms is expected to not change noticeably. The solid solution strengthening can be calculated using the following equation [45]:

$$\Delta \sigma_{ss} = \left( k_i^{1/n} C_i \right)^n$$  \hspace{1cm} (1)

where $C_i$ is the concentration of the solute atoms, $n$ is taken as 2/3, and $k_i$ is a factor related to individual solute elements, which is taken as 40 MPa (at.%)$^{-2/3}$ for Zn [46]. Then, we can calculate the strength provided by the Zn solid solution, as 9.2 MPa, 14.4 MPa, and 18 MPa for extruded alloy 2, alloy 3, and alloy 4, respectively. Thus, the solution strengthening due to Zn addition is not as obvious as the fine grain strengthening. In this work, the improvement in both strength and ductility of the Mg-2Al-0.5Ca alloy sheet is achieved through 0.6 wt.% of Zn addition. Compared to the AZ series alloy [47,48], the Mg-2Al-0.5Ca-0.6Zn alloy shows improved mechanical properties, with a relatively low content of alloying additions. This study is expected to contribute to the development of promising strong, ductile, and low-cost Mg alloy sheets.

5. Conclusions

In this study, the microstructure, texture, and mechanical properties of as-extruded Mg-2Al-0.5Ca-xZn alloys with different Zn additions were investigated. It was found that grain size was refined and texture weakening was achieved with the Zn addition in the Mg-2Al-0.5C alloys. The addition of 0.6 wt.% Zn improved the tensile strength and ductility of the as-extruded Mg-2Al-0.5Ca alloy sheet, which showed a 0.2% proof stress of 145 MPa, an ultimate tensile strength of 316 MPa, and an elongation of 30.0% along the extruded direction. The combination improvement of strength and ductility was mainly due to the fine and homogeneous grain microstructure and the weakened ED-
tilted texture. The as-extruded sheet showed little in-plane anisotropic tensile properties. The 0.2% proof stress, ultimate tensile strength, and elongation in the 45° direction were 148 MPa, 299 MPa, and 25.0%, and those in the transverse direction were 148 MPa, 269 MPa, and 16.8%, respectively.

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