The microstructure evolution and mechanical properties improvement of the AZ61 alloy by adding Sc

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Abstract

The improvement of mechanical properties and the microstructure evolution through adding Sc to AZ61 magnesium alloy were studied. The results indicated that the Mg₁₇Al₁₂ phase in the extruded AZ61 alloy was mainly distributed around the sub-structured and fine deformed grains, resulting in the nonuniform microstructure. The addition of Sc could effectively suppress the band-like precipitation of Mg₁₇Al₁₂ phase and improve the uniformity of microstructure. The grain sizes of the extruded alloys showed a trend of first decreasing and then increasing with the increase of Sc, which was mainly attributed to the secondary phase. The AZ61–0.5Sc alloy exhibited the best mechanical properties, its ultimate tensile strength and yield strength were 14.8 MPa and 40.8 MPa higher than those of the extruded AZ61 alloy, respectively, which was ascribed to the fine grains and abundant secondary phase in the alloy.

1. Introduction

The magnesium and its alloys have huge potential for the development in the automotive, aircraft, aerospace, medical implant and 3C industries due to low density, good cast-ability, and high specific strength and specific stiffness [1–3]. Considering the efficiency and cost of mass production of complex, thin-walled and near-net-shape components, the most of the magnesium alloys are produced by high-pressure die casting [4–6]. However, compared with wrought magnesium alloy, the casting magnesium alloy and die-casting magnesium alloy have disadvantages such as low strength, poor ductility, and limited processing properties at room temperature. Extruded magnesium alloy as a typical wrought magnesium alloy possesses more excellent mechanical properties, and improves the safety of the engineering components. Therefore, extruded magnesium alloy has potential application value as a structural material.

Up to now, a lot of the researches are mainly Mg–Al [7, 8], Mg–Zn [9, 10], and Mg–RE [11, 12] system alloys. In the Mg–Al system alloys, AZ61 alloy is widely used owing to excellent casting properties and low cost. However, because Mg₁₇Al₁₂ phase is the main strengthening phase in AZ61 alloy, the mechanical properties of AZ61 alloy are affected by its morphology, quantity and distribution, and its melting point is low, which leads to a mechanical properties degradation of the AZ61 alloy at elevated temperatures [13]. Therefore, it is feasible to improve the mechanical properties of the AZ61 alloy by controlling the morphology, quantity and distribution of the Mg₁₇Al₁₂ phase through alloying. Adding rare earth elements to magnesium alloy can not only refine the grains, purify the alloy [14, 15], and form the intermetallic compounds with a higher melting point than that of the Mg₁₇Al₁₂, which leads to the increase in strength of the alloy at the room temperature and elevated
temperatures, but also promote the dynamic recrystallization (DRX) of the alloy during hot deformation, which results in the weakened deformation texture and the improved ductility of the alloy as well [16].

Previous investigations indicated [17–20] that the addition of minor Sc could refine grains of the magnesium alloy and form the Sc-containing precipitates, which improved the mechanical properties of the magnesium alloy. Therefore, Sc is chosen to further enhance the mechanical properties of AZ61 alloy. Compared with other rare earth elements such as Ce, Nd, Y, and Gd in magnesium alloy, Sc has higher solid solubility in magnesium (15.9%) and lower density (3 g cm\(^{-3}\)), which implies that adding Sc in AZ61 alloy can not only improve the mechanical properties, but also maintain the low density of magnesium alloy. In previous reports, most studies have been conducted on Mg–Sc binary alloy. Ogawa et al. [21] found that adding 20at% Sc to pure Mg, the c/a value of \(\alpha\)-Mg matrix was reduced from 1.624 to 1.615, which led to the increasing of the tensile elongation. In addition, several studies have also investigated the precipitation hardening behavior of Mg–Sc binary alloy [22], shape memory effect [23], Liu et al. [24] prepared biodegradable bone. Due to the high price of Sc element, the application of Mg–Sc binary alloy is limited. Therefore, it is more practical to add minor Sc in magnesium alloy. Xiao et al. [19] found that adding 0.3wt% Sc to AZ91 alloy could refine the grains of AZ91 alloy and optimize the morphology and distribution of Mg\(_{17}\)Al\(_{12}\) phase, thereby improving the mechanical properties. Lin et al. [25] found that minor Sc addition could suppress the formation of Mg\(_{17}\)Al\(_{12}\) phase in AZ91 alloy, refine and modify Mg\(_{17}\)Al\(_{12}\) phase, promote the formation of continuous precipitation (CP) phase during aging, and simultaneously suppress the formation of discontinuous precipitation (DP) phase, which was conducive to aging strengthening. However, the researches on the effects of Sc on wrought magnesium alloy are very limited in the previous reports, especially AZ61 alloy (to the knowledge of the authors). Therefore, in this paper, the effects of Sc on the microstructure and mechanical properties of the extruded AZ61 magnesium alloy are investigated.

### 2. Experimental procedure

| Table 1. Nominal chemical compositions of the extruded alloys, wt%. |
|-------------------|-----|-----|-----|-----|
| Al    | Zn  | Sc  | Mg  |
| AZ61  | 6   | 1   | 0   | Bal. |
| AZ61–0.5Sc | 6   | 1   | 0.5 | Bal. |
| AZ61–0.8Sc | 6   | 1   | 0.8 | Bal. |
| AZ61–1.1Sc | 6   | 1   | 1.1 | Bal. |

Pure Mg (99.99%, wt%), Al (99.99%, wt%), Zn (99.99%, wt%), and Mg–20Sc (wt%) master alloy were used to prepare AZ61-xSc alloys through traditional melting casting method. Before casting, magnesium ingots were polished to remove the oxide film on the surface. Under the protection of Ar and SF\(_6\) (99:1) atmosphere, pure Mg was melted in a low carbon steel crucible in the resistance furnace. When the melt temperature rises to 750 °C, Mg–20Sc master alloy, pure Al and pure Zn were added in sequence. The melt was fully stirred, and the scum on the surface of the melt was removed after all metals were melted. Subsequently, the temperature was lowered to 730 °C and kept for 30 min to ensure sufficient and uniform diffusion of solute atoms. Then, the alloy melt was cast into a mold preheated at 300 °C to obtain an ingot with a size of \(\phi\) 60 mm \(\times\) 120 mm. The ingots were turned into round billets with a size of \(\phi\) 46 mm \(\times\) 70 mm. These round billets were homogenized at 420 °C for 16 h and then water-cooled to room temperature. Before extrusion, the homogenized round billets and the die were preheated at 380 °C for 30 min in the muffle furnace to reach the extrusion temperature. The preheated billets were extruded by IM-Y300 four-column hydraulic press, the extrusion temperature was 380 °C with an extrusion ratio of 13:1, and then air-cooled to room temperature. The bars with a diameter of 12.8 mm were obtained after hot extrusion. The extruded bars were turned to prepare standard tensile samples. The nominal chemical compositions of the extruded alloys are listed in table 1.

The x-ray diffraction (XRD, Bruker X8 ADVANCE A25) and transmission electron microscopy (TEM, JEM-2100F) were used to accurately analyze the phase of the alloys. The microstructure of the alloys was observed by optical microscopy (OM, LEICA DMI3000M) and electron backscatter diffraction (EBSD). The field-emission scanning electron microscopy (SEM, Zeiss ZEISS-6035) equipped with Oxford energy dispersive x-ray spectrometer (EDS) was used to analyze the morphology, quantity, distribution and element composition of the secondary phase. The metallographic samples were prepared by standard processes of grinding, polishing and etching, and the etching solution included 2.1 g picric acid, 35 ml ethanol, 5 ml distilled water and 5 ml...
acetic acid. The extruded bars were cut into thin slices with a thickness of 0.7mm to prepare TEM samples. The thin slices were ground to approximately 40 μm and then ion-polished to perforation. Finally, an universal testing machine (Instron-5982) was used to test the mechanical properties of the alloys at room temperature, the tensile rate was 0.1 mm min⁻¹, and the load was along the extrusion direction (ED).

3. Results

3.1. The phase analysis

Figure 1 shows the XRD patterns of the extruded AZ61-xSc alloys. It is observed that the alloys are mainly consisted of α-Mg and β-Mg₁₇Al₁₂ phases. The Al₃Sc is formed in the Sc-containing alloys, but they show lower peak intensity, indicating that the content of the Al₃Sc is low. With the increase of Sc content, the diffraction peaks of the Mg₁₇Al₁₂ phase are gradually weakened, which means that Mg₁₇Al₁₂ gradually decreases, and when the Sc content reaches 1.1wt%, the Mg₁₇Al₁₂ phase cannot be detected.

The TEM observation was conducted to further confirm the secondary phase of the alloys. Figure 2 shows the bright-filed TEM images and selected area electron diffraction (SAED) of the extruded AZ61–0.5Sc alloy. There are two different precipitates in the alloy, one is lamellar-like particles, and the other is irregular particles,
as shown in figures 2 (a) and (d), respectively. The high-resolution images and SAED results confirm that the lamellar precipitation is Mg17Al12, and the irregular precipitation is Al3Sc. Furthermore, the TEM-EDS results also suggest that the stoichiometric composition of the irregular particles is Al3Sc. Combined with the XRD results, there are mainly two secondary phases in AZ61-xSc alloys: Mg17Al12 and Al3Sc.
3.2. The microstructure evolution and mechanical properties

Figure 3 shows the SEM images of the extruded AZ61–0.5Sc alloy. It is observed that there are two different morphologies of the precipitates. The one having a fine particle morphology forms a band parallel to extrusion direction, and the other has a coarse regular polygon morphology. Table 2 lists the EDS results of each point in figure 3. Combined with XRD, TEM and EDS results, the finer precipitates are Mg17Al12, and the coarser precipitates are Al3Sc. Most of the fine Mg17Al12 particles distribute along the grain boundaries and a few of them exist within the intragranular. The coarse Al3Sc particles distribute at grain boundaries.

Figure 4 shows the OM images of the extruded AZ61-Sc alloys. It can be observed that there are the fine equiaxed grains and coarse deformed grains in the alloys. With the increasing of Sc, the deformed grains gradually decrease, which indicates that adding Sc to AZ61 alloy has effect on the DRX behavior during extrusion. The extruded AZ61 alloy without Sc contains lots of precipitate bands along the extrusion direction (figure 4 (a)). This result is similar to the previous studies [26, 27]. The precipitate bands gradually decrease with the increase of Sc, and when the Sc content reaches 1.1wt%, they are almost not observed. According to figure 3 and table 2, it is found that the precipitate bands are mainly Mg17Al12. Figure 5 shows the equilibrium phase diagram of the Mg–6Al–xSc alloys. The phase diagram is used to analyze the precipitation behavior of Mg17Al12 phase in AZ61-xSc alloys during extrusion. It is obvious that the Mg17Al12 phase in the Mg–6Al–xSc (x = 0, 0.5, 0.8, 1.1, wt%) alloys is precipitated below 300 °C. Since the extrusion temperature of 380 °C is located in the α-Mg and Al3Sc phases region, the alloys hardly undergo dynamic precipitation to form Mg17Al12 during extrusion. When the extruded bars are air-cooled after exiting the extrusion mold, they will undergo the static precipitation to form Mg17Al12 owing to that the temperature reduction from 380 °C to room temperature leads to a decrease in the solubility limit of Al atoms in the α-Mg matrix during the air cooling stage. Therefore, the main reasons for the decrease of the precipitate bands in the Sc-containing extruded alloys are that Sc suppresses the static precipitation of the Mg17Al12. Furthermore, adding 0.5wt% Sc to AZ61 alloy, the grains are obviously

![Figure 5. Equilibrium phase diagram of the Mg–6Al–xSc (x = 0–1.5wt%).](image_url)

![Figure 6. Mechanical properties of the extruded alloys: (a) tensile engineering stress-strain curves; (b) corresponding YS, UTS, Elongation.](image_url)
refined. However, it is worth noting that as Sc content continues to increase, the grains are coarsened, which is attributed to the substantial reduction of the Mg17Al12.

Figure 6 (a) shows the tensile engineering stress-strain curves of the extruded AZ61-xSc alloys, the corresponding values are listed in figure 6 (b), including ultimate tensile strength (UTS), 0.2% yield strength (YS) and elongation (El). It is observed that adding 0.5wt% Sc to AZ61 alloy can effectively improve the mechanical properties of the extruded alloy. The UTS and YS of the extruded AZ61–0.5Sc alloy reach 327.5MPa and 225.8MPa, respectively, which are 14.8MPa and 40.8MPa higher than those of the extruded AZ61 alloy. Surprisingly, although the UTS and YS of the extruded AZ61–0.5Sc alloy are significantly improved, the elongation is not lost. However, as the Sc content continues to increase, the mechanical properties of the alloys gradually decrease, which is mainly ascribed to the influence of the grain sizes and the precipitates. The mechanical properties can be improved by the fine grains of the alloy. Although adding Sc to AZ61 alloy can suppress the formation of Mg17Al12, there is still a lot of Mg17Al12 in AZ61–0.5Sc alloy (figure 4 (b)), thus the Mg17Al12 might play an important role on improving the mechanical properties of the alloy. These fine particles can effectively pin the slip of the dislocations, and the Al3Sc phase can also hinder the dislocation motion, thus the extruded AZ61–0.5Sc alloy are significantly strengthened. The Mg17Al12 phase is greatly reduced by adding 0.8–1.1wt% Sc to AZ61 alloy, and the strengthening of the alloys through the rare Al3Sc phase cannot make up for the strength loss caused by the reduction of the Mg17Al12 phase, so the UTS and YS are decreased.

3.3. EBSD analysis
Figure 7 shows the microstructure distribution of the extruded AZ61-xSc alloys. Blue, yellow and red represent dynamically recrystallized (DRXed) grains, sub-structured grains and deformed grains, respectively. Most of the grains in the alloys are DRXed grains, which indicates that the alloys have fully recrystallized. A few sub-structured grains and deformed grains are distributed in band-like along the extrusion direction. Combined with figure 4, it is found that the precipitate bands in figure 4 are consisted of the fine sub-structured grains and deformed grains, and a lot of Mg17Al12 are distributed around them.

Figure 8 shows the inverse pole figure (IPF) maps and (0001) basal pole figures of the extruded AZ61-xSc alloys. As shown in figures 8(a), (c), (e) and (g), it is observed that the alloys are mainly the equiaxed grains, which is consistent with the OM images (figure 4). According to the EBSD results, the average grain sizes of the four alloys are 9.12 μm, 6.97 μm, 10.95 μm and 13.54 μm, respectively. Obviously, minor Sc addition can effectively refine the extruded AZ61 alloy grains. In addition, the color of the grains is randomly distributed, indicating that there is no obvious grain preference orientation in the alloys, so the texture of the alloys is relatively weak. As shown in figures 8(b), (d), (f) and (h), the extruded alloys present a typical fiber texture (the c-axis of the grains is perpendicular to the extrusion direction). In the extruded AZ61 alloy without Sc, the (0001) basal poles are distributed along the transverse direction (TD). After adding Sc, the (0001) basal poles of the Sc-containing
extruded AZ61 alloys are distributed on both sides of TD. Obviously, adding 0.5–1.1wt% Sc to AZ61 alloy causes the (0001) basal poles to rotate toward the ED. Similar phenomena exist in most of the rare earth magnesium alloy: adding the rare earth elements can cause the rotation of the basal pole of the deformed magnesium alloy [28, 29].

4. Discussion

The results mentioned above indicate that, in the extruded AZ61 alloy without Sc, a lot of Mg17Al12 phase are unevenly precipitated around the sub-structured grains and fine deformed grains during the cool-down stage after extrusion, forming the precipitate bands (figure 4 (a)), which causes the nonuniform microstructure. It is well known that the sub-structured grains and deformed grains do not undergo DRX, thereby storing much strain energy. Therefore, the Mg17Al12 preferentially nucleate and grow around these grains. According to the report of Li et al [30], the distribution of the Al atoms in the round billets is nonuniform even after they are homogenized, and due to the inhomogeneous flow of the metal, the strain imposed is also nonuniform during

![Figure 8. Inverse pole figure maps and (0001) basal pole figures of the extruded alloys: (a), (b) AZ61; (c), (d) AZ61–0.5Sc; (e), (f) AZ61–0.8Sc; (g), (h) AZ61–1.1Sc.](image)
extrusion [31]. Therefore, during the cool-down stage, the Mg17Al12 phase are locally formed in the areas where there is sufficient driving force to promote static precipitation (that is, the areas with high Al concentration or high internal strain energy). Finally, the locally precipitated Mg17Al12 phase forms a band parallel to the extrusion direction in the extruded AZ61 alloy. In addition, adding 0.5–1.1 wt% Sc to AZ61 alloy can suppress the static precipitation of Mg17Al12, effectively improving the microstructure of the Sc-containing extruded alloys. Because the radius of Sc atoms is relatively larger than that of the Al and Zn atoms [32], and the diffusion coefficient of Sc in α-Mg is low, Sc atoms will hinder the diffusion of Al atoms. According to the studies of Pan et al [32] and Xiao et al [19], Sc decreases the diffusion coefficient of Al atoms in solid solution, thereby causing the decreasing of the segregation of Al atoms at the grain boundaries. Therefore, it is inferred that the influence of Sc on the diffusion kinetics of Al atoms in the magnesium alloy may also be one of the reasons for the decrease of Mg17Al12 in the Sc-containing extruded AZ61 alloys. In addition, adding Sc to the AZ61 alloy forms a high-temperature stable Al3Sc phase. According to the Mg–Al–Sc phase diagram (figure 5), Al3Sc is formed in preference to Mg17Al12 during the solidification–cooling stage of the alloy, which implies that Al atoms preferentially combine with Sc atoms to form Al3Sc. The formation of Mg17Al12 is suppressed due to the lack of Al atoms. The Al3Sc distributed at the grain boundaries occupies the nucleation and growth position of Mg17Al12 phase. The nucleation and growth of the Mg17Al12 are suppressed, resulting in the decrease of the Mg17Al12 phase in the Sc-containing extruded AZ61 alloys.

5. Conclusions

Based on the experimental investigation on the microstructure and mechanical properties of the AZ61-xSc alloys, the conclusions can be drawn as follows:

1. In the extruded AZ61 alloy without Sc, the Mg17Al12 phase are mainly precipitated around the substructured grains and fine deformed grains, and then forming a lot of precipitate bands.

2. The addition of Sc can suppress the static precipitation of Mg17Al12 phase during the cool-down stage after extrusion, thereby improving the microstructure of the Sc-containing extruded alloys.

3. Adding 0.5 wt% Sc to AZ61 alloy can refine the grains and improve the mechanical properties. As Sc continues to increase, the grains are coarsened and the mechanical properties are decreased.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

Conflict of interest

Authors state no conflict of interest.

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