Study on Sc Microalloying and Strengthening Mechanism of Al-Mg Alloy

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Abstract: In this study, Al-Mg alloys were prepared by vacuum + argon-protected casting. The effects of Sc microalloying on the microstructure and mechanical properties of Al-Mg alloy were studied. The strengthening mechanism of Al-Mg alloy with Sc was discussed. The results show that the average grain size of as-cast Al-Mg alloy with Sc reaches 36.07 µm, which is 74.4% smaller than that of alloy 1 without refiner and 32.0% of alloy 2 with 0.15% Ti. The alloy with Sc has excellent comprehensive mechanical properties, with a tensile strength of 274 MPa and an elongation of 29.67%, and its tensile strength is 10.6% higher than alloy 1 and 7.9% higher than alloy 2. There exist two kinds of fine particles in as-cast Sc-containing Al-Mg alloy, nucleated particles Al3Sc, and dispersed second-phase particles Al3(Sc, Ti). The main strengthening mechanisms of the as-cast Al-Mg alloy with Sc reveal the solid solution strengthening of Mg and the fine-grain strengthening effect caused by Sc, accounting for 57.7% and 23.9% to strengthening contribution, respectively.

Keywords: Al-Mg alloy; Sc microalloying; microstructure; mechanical properties; strengthening mechanism

1. Introduction

Al-Mg alloy has excellent corrosion resistance, good welding performance and fatigue performance. It is often used as a welding filler material in the marine industry, in automobiles, high-speed trains, in the aerospace industry, and in other fields [1–3]. Al-Mg welding filler generally needs to meet the requirements of high-strength Al-Zn-Mg aluminum alloy welded joints for strength, plasticity, thermal cracking resistance, and corrosion resistance, etc. [4–6]. The strength of Al-Mg alloy is primarily from solid solution strengthening by Mg, which has a substantial solid solubility in aluminum [7,8]. However, with the increase of Mg content (above 3.5%), the β-Al3Mg2 phase will precipitate at the grain boundary, reducing the stress corrosion resistance and intergranular corrosion resistance of the alloy [9].

An effective approach to improve the strength of Al-Mg alloy is the addition of rare and transition elements as a microalloying element [10–12]. A trace amount of Ti can make the aluminum melt form a large number of tiny heterogeneous nucleus particles TiAl3 under a small degree of undercooling [13]. However, poisoning of Al−5Ti−B grain refiners was observed readily interact with other elements, resulting in grain refinement strengthening fading behavior [14]. Sc serves as a potent grain refiner in castings, which improves both the strength and formability [15]. Also, the fine dispersion of Al3Sc precipitates is known to inhibit recrystallization during thermo-mechanical processing [16]. Furthermore, the addition of Sc or Zr to welding filler alloy has been shown to have a beneficial effect on the weldability and thermal crack resistance of aluminum alloys [17,18]. However, some
coarse particles of Al₃(Sc, Zr) that are not L12 structure are generated, and the coherence relationship with the Al matrix is poor [19].

Owing to the great contribution of Sc grain refinement, Al₃Sc primary particles have been extensively studied by many researchers [20,21]. Norman et al. [21] have suggested that primary Al₃Sc particles may have a cusped cubic morphology. N. Kishore Babu et al. [22] have also noted the morphology of primary particles appeared on the grains center and grain boundaries nearby by means of metallographic observation. However, dedicated investigation on quantitative analysis of the relationship between microstructure and properties is scant, and the underlying strengthening mechanism of grain refinement remains unclear.

The aim of this study is to study the effects of Sc microalloying on the microstructure and properties of as-cast Al-Mg alloys, wherein Al-Mg alloys with different compositions are prepared by vacuum + argon-protected casting. The transmission electron microscopy (TEM) is used to further reveal the refined phase structure. The strengthening effect of Sc-containing Al-Mg alloy is quantitatively analyzed. On this basis, the main strengthening mechanism of as-cast Al-Mg alloys will be discussed.

2. Materials and Methods

The experimental Al-Mg alloys were prepared by pure Al (99.99% purity, all in wt.%), Mg (99.99%), Al-10%Mn, Al-10%Cr, Al-2%Sc, and Al-5%Ti-1%B master alloys. Table 1 shows the chemical composition of Al-Mg alloys designed with different compositions.

Table 1. Chemical compositions of the Al-Mg alloy designed with different compositions (wt.%).

| Alloy | Mg  | Mn  | Cr  | Ti  | Sc  | Al  |
|------|-----|-----|-----|-----|-----|-----|
| Alloy 1 | 5.0 | 0.15 | 0.15 | -   | -   | Bal.|
| Alloy 2 | 5.0 | 0.15 | 0.15 | 0.15 | -   | Bal.|
| Alloy 3 | 5.0 | 0.15 | 0.15 | 0.15 | 0.15 | Bal.|

Al-Mg alloy ingots were prepared by vacuum + argon-protected casting. Before smelting, the pure aluminum, Al-10%Mn, Al-10%Cr, and Al-5%Ti-1%B were first put into the graphite crucible in the vacuum furnace, and then the casting system was placed in the vacuum melting furnace, as shown in Figure 1. The smelting and casting processes were as follows. Firstly, a vacuum was created to $9.0 \times 10^{-2}$ through a mechanical pump, roots pump, and diffusion pump in turn, and then pure aluminum and master alloys were heated at a maximum temperature of 800 °C. When the smelting was completed, the vacuum was removed, and argon was filled at 390 Pa. Al-2%Sc and pure magnesium were then added in the melt through the feeding system sequentially, where the temperature of adding magnesium was 730 °C. In order to prevent magnesium from floating and burning, a stirring blade with a shape of immersion bell was used. After the alloy melt was held at 710 °C for 10 min, the melt was poured into a metal mold and solidified into the Al-Mg alloy ingot of Φ120 × 420 mm.

Metallographic observation was conducted for the as-cast samples on the transverse section using an Axiovert 200 MAT ZEISS optical microscope. The samples were prepared following the polishing procedure and anodic coating using Bake’s reagent composed of 5 mL HBF₄ and 195 mL H₂O, with an electroplating voltage of 30 V and a coating time of 45 s. The average grain size was calculated according to the intercept method. The morphology of the second phase was observed by JSM. 7001 F scanning electron microscope (SEM). The elemental distribution of the second phases were analyzed by JSM. 7001 F scanning electron microscope (SEM) with EDS for point scanning. D8 advance X-ray diffractometer (XRD) was adopted for phase identification and lattice constant calculation, and the precipitates were observed by Tecnai™ transmission electron microscopy (TEM). The alloy tensile test specimens were processed according to the standard of GB/T228-002 “Metal Tensile Test Specimen”, as shown in Figure 2. The tensile test was carried out on the CSS-4100 electronic universal material testing machine with a strain rate of $1.5 \times 10^{-3}$ s⁻¹.
3. Results

3.1. Microstructure of As-Cast Al-Mg Alloys

Figure 3 shows the metallography of the as-cast Al-Mg alloys designed with three different compositions. The fully equiaxed grains can be seen in these alloys. The grains present different sizes due to different compositions, and the grain size of alloys were measured and exhibited in Figure 4. Alloy 3 has the smallest grain size, and the average grain size is 36.07 µm, which is 74.4% smaller than that of alloy 1 and 32.0% of alloy 2. Alloy 2 added with 0.15% Ti also has a good grain refinement effect, which is consistent with findings of previous workers [13], and the as-cast alloy 2 with an average grain size of 53.06 µm is 62.4% less than that of Alloy 1.
Figure 3. The microstructure of as-cast Al-Mg alloys with different compositions: (a) alloy 1; (b) alloy 2; (c) alloy 3.

Figure 4. The average grain size of as-cast Al-Mg alloys with different compositions.

3.2. Analysis of Second Phase

Figure 5 exhibits the SEM images of the Al-Mg alloys with three different compositions. As can be seen from Figure 5a,c,e, with the designed addition of Ti and Sc to the Al-Mg alloy, the density of the second phase particles increases, and the micro-alloying effect of Al-Mg alloy is enhanced. Figure 5b,d,f show the point scan analysis results of the second phase of alloys 1, 2, and 3, respectively. The second phase in alloy 1 is Al$_6$Mn, which is small in size and low in content. There are two kinds of morphological second phases in alloy 2 including the coarse second phase and the fine second phase. The coarse second phase is Al-Mg-Mn-Cr-Ti compounds (Points 3 and 4 in Figure 5d), indicating that the poisoning effect of Ti addition is obvious, and the results were found by Mohantyg [23]. The fine second phase is determined according to the spot scan results as for Al$_3$Ti particles. Spot scan results of alloy 3 are similar to those of alloy 2 shown in Figure 5f; the coarse phase is the Al-Mg-Mn-Cr-Ti-Sc compound, and the fine phase may include dispersed particles of Al$_3$Sc or Al$_3$(Sc, Ti).
In order to determine the composition of the fine precipitates in the alloy 3, the precipitates were observed by TEM. Figure 6 shows a TEM photograph and a diffraction pattern of the precipitation phase of the alloy 3. Two kinds of precipitation phases with different sizes were observed; the larger phase size was about 200 nm, and the fine phase size was about 8.9 nm. According to the analysis results of the diffraction pattern Figure 6b of the massive precipitated phase in Figure 6a, it is confirmed that the phase is Al$_3$(Sc, Ti) phase, and the core is Al$_3$Sc (structure surrounded by red lines), and the outer layer is Al$_3$Ti (structure surrounded by green lines). The epitaxially grown Al$_3$Ti is no longer the D0$_{22}$ structure, but maintains the Al$_3$Sc metastable L1$_2$ structure, and there exists a certain amount of dislocation around the coarse phase as shown Figure 6a. Also, a small amount of fine primary Al$_3$Sc phase existing in the alloy shown in Figure 6c, which can be the heterogeneous nucleation sites of grains and subgrains.
3.3. Mechanical Properties of As-Cast Al-Mg Alloys

Figure 7 depicts the room temperature engineering stress-strain curves of as-cast Al-Mg alloys, and the tensile mechanical properties results are exhibited in Figure 8. The three curves in Figure 7 are all zigzags, which is a phenomenon of plastic instability (Portevin-Le Chatelier) [24]. The stress-strain curve of alloy 3 is higher than that of the Alloy 1, 2, and has a higher specified plastic extension strength of 114.3 MPa and tensile strength of 274 MPa. The specified plastic extension strength of alloy 3 is 19.1% higher than alloy 1 and 5.8% higher than alloy 2. The tensile strength of the alloy 3 increased by 10.6% compared with alloy 1 and increased by 7.9% compared with alloy 2. However, the elongation rate of alloy 3 is slightly decreased. The probable explanation is that the high density of the second phase in Alloy 3 shown in Figure 5e, which produces a high precipitation strengthening effect and, at the same time, slightly reduces the elongation of the alloy.

Figure 6. Microstructure TEM images of Al-Mg Alloy with Sc: (a) bright field image of Al$_3$(Sc, Ti) phase; (b) Al$_3$(Sc, Ti) phase diffraction pattern along the <001> crystallographic direction of Al matrix; (c) bright field image of Al$_3$Sc phase; (d) Al-Mg alloy high-resolution image; (e) high-resolution image of Al$_3$Sc phase; (f) FFT of (e).
where \( \sigma_{\text{ss}} \) is the Peierls stress caused by lattice mismatch of high-purity metal, and for the Al-Mg alloy, the value of \( \sigma_0 \) is 19.2 MPa [26].

The solid solution strengthening effect is positively related to the concentration of solid solution atom Mg in Al-Mg alloy, which can be expressed by Formula (2) [25]. The solid solution content of Mn and Cr is small, and the strengthening effect can be ignored.

\[
\sigma_{\text{ss}} = HC_{\text{Mg}}
\]  

(2)

where \( H \) is a constant, \( C_{\text{Mg}} \) is the concentration of solid solution Mg in the matrix, \( n \) is a constant related to the type of solid solution, and the values of \( H \) and \( n \) are shown in Table 2. Figure 9 shows the X-ray diffraction patterns of alloys with different compositions. It can be seen that the main phase is \( \alpha \)-Al solid solution in Al-Mg alloy. The diffraction peaks of Al-5Mg, Al-5Mg-0.15Mn, Al-Mg Alloy without Sc and Al-Mg Alloy with Sc all deviates significantly from the diffraction peak of pure aluminum. However, the solid

**4. Discussion**

In the above, the microstructure and mechanical properties of the Sc microalloying as-cast Al-Mg alloy could be greatly improved. In order to clarify the relationship between the microstructure and properties, the strengthening mechanism of the as-cast Al-Mg alloy with Sc will be studied. The yield strength of the alloy depends on the number of defects that need to be overcome during the tensile process. The strengthening mechanisms of as-cast Al-Mg alloys include solid solution strengthening \( (\sigma_{\text{ss}}) \), grain boundary strengthening \( (\sigma_{\text{GB}}) \), and precipitation strengthening \( (\sigma_p) \). The yield strength \( (\sigma_y) \) can be expressed by Formula (1) [25].

\[
\sigma_y = \sigma_0 + \sigma_{\text{ss}} + \sigma_{\text{GB}} + \sigma_p
\]  

(1)

\[\sigma_{\text{ss}} = HC_{\text{Mg}}\]  

(2)
solution content of Mn, Cr, Ti and Sc is small, and the change of the lattice constant can be ignored. It is shown that the solid solution of Mg element leads to the change of the lattice constant of the aluminum matrix. Some studies have shown that the lattice constant of Al increases by $4.6 \times 10^{-4}$ nm for every 1 at% increase in Mg [27–29]. The lattice constant of the solid solution can be calculated according to the XRD test results, and the calculation results of solid solubility are shown in Table 3. The solid solubility of Mg in the Sc Al-Mg alloy is 4.56%, and the contribution of the solid solution strengthening of Mg to the yield strength is calculated to be 68.2 MPa by Formula (2).

Table 2. Coefficients in the strength calculation formula [26,29,30].

| Parameter | $\sigma_0$/MPa | $H$/MPa (wt.% Mg)$^{-n}$ | $n$ | $k$/MPa·m$^{1/2}$ | $G$/GPa | $b$/nm |
|-----------|----------------|--------------------------|-----|------------------|---------|--------|
| Value     | 19.2           | 12.1                     | 1.14| 0.17             | 26.9    | 0.286  |

Note: $\sigma_0$ is Peierls stress, $k$ is Hall-Petch coefficient, $G$ is shear modulus, $b$ is Burger’s vector, and $H$ and $n$ are constants.

![Figure 9. XRD patterns of alloys with different compositions.](image)

Table 3. XRD lattice parameter detection and solid solubility calculation results.

| Alloy                  | Lattice Parameter/nm | Crystal Indices | Solid Solution Concentration/% |
|------------------------|----------------------|-----------------|-------------------------------|
|                        |                      | (111)           | (200)                         |
| Aluminum               | $d$                  | 2.3379          | 2.0247                        |
|                        | $a$                  | 0.40494         | 0.40494                       |
|                        | Average value of $a$ | 0.4049          |                               |
| Al-5Mg                 | $d$                  | 2.3503          | 2.0349                        |
|                        | $a$                  | 0.40708         | 0.40698                       |
|                        | Average value of $a$ | 0.4070          | 4.56                          |

The rare earth element Sc exists in alloy 3 in the form of primary Al$_3$Sc, which can be used as a heterogeneous nucleation site for aluminum matrix, and has the great effect of refining grains. According to the Hall-Petch Formula (3) [28,29], the fine-grain strengthening effect in the Al-Mg alloy can be calculated.

$$\sigma_{GB} = k d^{1/2}$$

where $k = 0.17$ MPa·m$^{1/2}$ is Hall-Petch coefficient. According to the calculation results in Figure 4, the average grain size is 36.07 μm, and the yield strength increment caused by grain boundary strengthening is 28.3 MPa.

The size of Al$_3$(Sc, Ti) phase in alloy 3 is about 200 nm, and the main strengthening mechanism is Orovan strengthening by bypassing the phase. Under this mechanism, the
dislocations will bypass the precipitation phase and form a dislocation ring around the precipitation phase, which increases the dislocation density and improves the strength of the alloy. Its contribution to yield strength can be calculated from Formula (4) [30]:

\[
\sigma_p = \sqrt{3} G b / l
\]

where \( G \) is the shear modulus, \( b \) is the Burgers vector, and \( l \) is the spacing of the second phase particle. According to the SEM detection results, \( l = 8.9 \times 10^3 \) nm, and the yield strength increment of precipitation strengthening is 2.59 MPa.

Figure 10 shows a comparison diagram of the experimental yield strength and the theoretical calculation yield strength. It can be seen that the yield strength values of the two are roughly consistent. Due to certain errors in the calculation, the difference between the two can be ignored. The above theoretical calculation process can be used to predict the yield strength of Al-5Mg alloys. Correspondingly, the main strengthening mechanism of the alloy are the solid solution strengthening of Mg and the fine-grain strengthening effect caused by the Sc element, accounting for 57.7% and 23.9%, respectively. The combined effect of solid solution strengthening and fine-grain strengthening increases the resistance of dislocation movement of the alloy during the tensile mechanical test, thereby increasing the yield strength of the alloy.

![Figure 10. Comparison of calculated and measured yield strength of as-cast Al-Mg alloy with Sc.](image)

5. Conclusions

Three Al-Mg alloys with different compositions were prepared by vacuum + argon-protected casting. The effect of Sc on the microstructure and properties of as-cast Al-Mg alloy was studied, and its strengthening mechanism was discussed. The results are as follows:

1. As-cast Al-Mg alloy with 0.15% Sc has obvious grain refinement. The average grain size is 36.07 \( \mu \)m, which is 74.4% smaller than that of alloy 1 without refiner and 32.0% of Alloy 2 with 0.15% Ti;
2. The alloy with Sc has the highest tensile strength of 274 MPa, which is 10.6% higher than alloy 1 and 7.9% higher than alloy 2.
3. The main strengthening mechanism of the as-cast Al-Mg alloy with Sc reveals the solid solution strengthening of Mg and the fine-grain strengthening effect caused by the Sc, accounting for 57.7% and 23.9% to strengthening contribution, respectively.

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