

Article

Effects of Sc Microalloying on Microstructure and Properties of As-Extruded Al-5Mg Alloy

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Abstract: Al-Mg alloys were fabricated by hot extrusion process after vacuum + argon-protected casting. The effects of Sc microalloying on microstructure and properties of as-extruded Al-5Mg alloy were studied. The results show that alloy 2 containing Sc is partially recrystallized during hot extrusion, resulting in the structure of alloy 2 consisting of 21.8% equiaxed recrystallized structure and 78.2% fine banding structure. The grain size of alloy 2 is about 5.31 μm , which is 83.3% finer than that of alloy 1 without Sc. The sum of the yield strength increments due to grain boundary strengthening (σ_{GB}) in alloy 2 is 103.59 MPa, where low angle grain boundary strengthening (σ_{LAGB}) is increased by a factor of 4.5, and high angle grain boundary strengthening (σ_{HAGB}) is increased by 73.4% compared to that of alloy 1. The addition of 0.2% Sc leads to an increase in the lattice strain and dislocation density of alloy 2, and the dislocation strength (σ_{dis}) increases to 41.85 MPa. The alloy 2 with Sc has a higher tensile strength of 380.7 MPa, which is 34.1% higher than that of alloy 1, but alloy 2 has a 46.2% reduction in elongation compared to alloy 1.

Keywords: as-extruded Al-5Mg alloy; Sc microalloying; microstructure; dislocation strengthening; grain boundary strengthening



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1. Introduction

High strength is an essential requirement for aluminum alloys applied in the fields of railway transportation, aerospace industry, military industry, etc. [1–3]. In recent years, the same requirement has been put forward for welding filler materials for welding high-strength aluminum alloys [4–6]. Since the preparation of welding fillers is a long-term process, including extrusion, drawing, rolling, etc., an increase in strength is often accompanied by processing difficulties [7].

Al-Mg alloys are usually preferred as welding fillers for high-strength aluminum alloys due to their suitable weldability and strength [8–10]. A combination of high strength and suitable formability in non-heat treatment strengthening Al-Mg alloys can be achieved by refining the grain size and introducing precipitates in the microstructure. For these purposes, some alloying elements such as Mn, Cr, Zr, and Sc are added to the aluminum alloys [11–13]. The effect of Mn and Cr in inhibiting recrystallization is limited, and it is particularly easy to generate a coarse second phase, which affects the processability of the Al-Mg alloys [14]. Element Zr has an obvious anti-recrystallization effect in Al-Mg alloys, where exist the coarse cuboidal Al_3Zr and the elongated wider grains leading to processing difficulties [15]. Element Sc addition is unique in aluminum alloy, where it may form an equilibrium, thermally stable, coherent Li_2 phase (Al_3Sc) with Al, which can be extremely effective in dislocation pinning, thus providing a high strengthening effect [16]. At the same time, the addition of Sc contributes to inhibiting recrystallization by increasing

the recrystallization temperature of the alloy [17]. The effects of Sc microalloying on the microstructure and properties of as-cast Al-Mg alloys has been studied, and dedicated investigation on quantitative analysis of the relationship between microstructure and properties is well known [18]. However, Al-Mg alloy containing Sc is partially recrystallized during hot extrusion, and its uneven microstructure has a great effect on the extrudability in Al-Mg alloys and the retarded recrystallization. No research work has been performed to explore the corresponding relationship between microstructure and properties.

The aim of this work is to study the effects of Sc microalloying on the microstructure and properties of as-extruded Al-5Mg alloys. The electron backscatter diffraction (EBSD) and X-ray diffractometer (XRD) analysis are carried out to explore the mechanism of anti-recrystallization in the as-extruded Al-5Mg alloys. The grain boundary strengthening and dislocation strengthening caused by the microalloying element Sc are quantified, whose contributions to the yield strength of the as-extruded Al-5Mg alloys are discussed.

2. Materials and Methods

Commercially pure aluminum, pure magnesium, Al-10%Mn, Al-10%Cr, Al-2%Sc, and Al-5%Ti-1%B master alloys were chosen as the raw materials. The practical chemical compositions of the Al-5Mg alloys were measured by a FOUNDRY-MASTER Pro electric spark direct reading spectrometer, as shown in Table 1. Al-5Mg alloy ingots were prepared by vacuum + argon-protected casting process [18]. Moreover, the Al-5Mg alloy ingots were extruded into the $\Phi 9.5$ mm wire on a 1250T FCSX-01 extruder after annealing at $480\text{ }^{\circ}\text{C} \times 10\text{ h}$, where the extrusion temperature was $490\text{ }^{\circ}\text{C}$, and the extrusion speed was 0.3–0.5 mm/s.

Table 1. Practical chemical compositions of the Al-5Mg alloys in this study (wt.%).

Element	Mg	Mn	Cr	Ti	Sc	Si	Fe	Al
Alloy 1	4.98	0.167	0.065	0.110	-	0.0111	<0.0001	Bal.
Alloy 2	5.02	0.166	0.067	0.112	0.2	0.0116	<0.0001	Bal.

Statistical analysis was performed on those microstructure features such as grain size distribution, grain boundary characteristics, and microstructures with the help of the EBSD technique. EBSD analysis was carried out on a JSM. 7001F scanning electron microscope (SEM) furnished with an Oxford EBSD analysis system. D8 ADVANCE X-ray diffractometer (XRD) was used to measure the diffraction peak and the half-peak width, where the scanning rate was set to $0.5\text{ }(^{\circ})/\text{min}$, the scanning range was $20\text{--}90^{\circ}$, the wavelength of Cu-K α was 0.154056 nm. The tensile sample is taken along the tensile direction of the extruded wire billet, wherein the parallel length of the cylindrical tensile specimen is 35 mm, and the diameter is 5 mm. The tensile test was carried out on the CSS-4100 electronic universal material testing machine with a strain rate of $1.5 \times 10^{-3}\text{ s}^{-1}$.

3. Results and Discussion

3.1. EBSD Analysis and Grain Boundary Strengthening

The microstructure characteristics of the two categories of Al-5Mg alloys after hot extrusion are shown in Figure 1. The microstructure of as-extruded alloy 1, as shown in Figure 1a, fully consists of fine, equiaxed grains with an average grain size of about $31.75\text{ }\mu\text{m}$ (Figure 1e and Table 2). By ranking the grain orientation spread (GOS) (Figure 1c), recrystallized structure, substructure, and deformed structure were identified. There exist 74.3% recrystallized grains, indicating that nearly complete dynamic recrystallization occurred in alloy 1. Figure 1b demonstrates the microstructure of the inverse pole figure (IPF) of as-extruded alloy 2, and there is a remarkably fine banding structure along the extrusion direction. Figure 1d shows the banding structure as a combination of an initially deformed structure and a substructure, accounting for 33.9% and 44.3%, respectively.

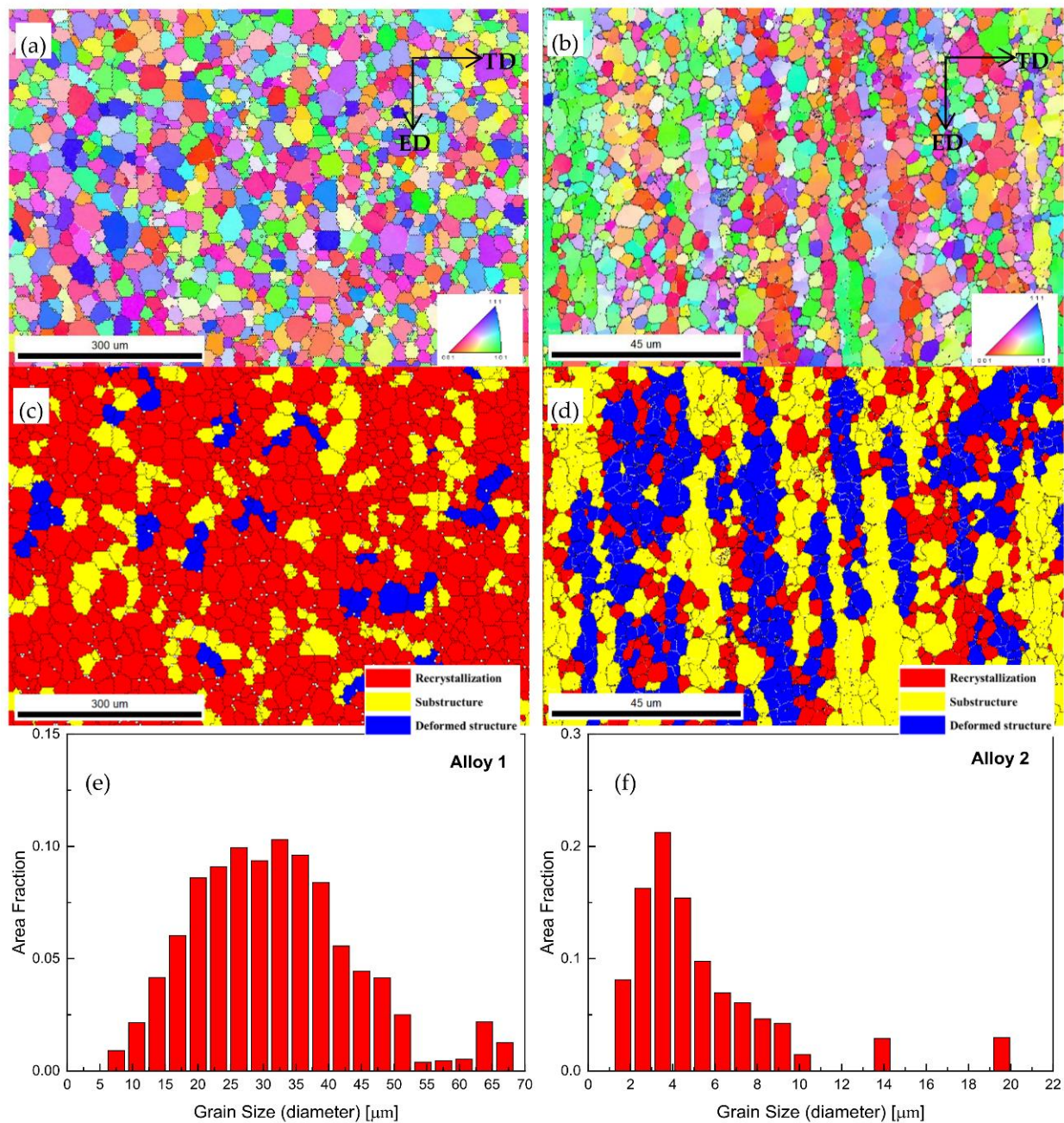


Figure 1. EBSD analysis of alloy 1 (a,c,e) and alloy 2 (b,d,f) after hot extrusion: (a,b) IPF; (c,d) GOS; (e,f) grain size distribution.

Table 2. Comparison of statistical microstructure characteristics for two categories of extruded Al-5Mg alloys.

	Recrystallization/%	Substructure/%	Deformed Structure/%	Average Grain Size (d)/μm
Alloy 1	74.3	18.5	5.9	31.75
Alloy 2	21.8	44.3	33.9	5.31

The alloy 2 with a Sc content of 0.2% can form many Al_3Sc dispersoids to inhibit dynamic recrystallization, resulting in only 21.8% recrystallized structure in extruded alloy 2. This is consistent with previous findings that the addition of Sc has an anti-recrystallization effect in aluminum alloys [19]. The microstructure of alloy 2 shown in

Figure 1b is much finer than that of alloy 1, even though the banding structure and the average grain size of alloy 2 are greatly decreased to about 5.31 μm .

Figure 2 shows the grain boundary misorientation (GBM) maps of as-extruded Al-5Mg alloys with different compositions. As shown in Figure 2a,b, the green line (θ between 2° and 15°) represents low angle grain boundary (LAGB), and the blue line ($\theta \geq 15^\circ$) represents high angle grain boundary (HAGB). The number fraction of LAGB in alloy 1 is extremely low at only 6.2%, while the number fraction of HAGB reaches 93.8%, as listed in Table 3. The formation of HAGB is evidence of a dynamic recrystallization mechanism. Therefore, it can be inferred that recrystallization has maturely occurred during the extrusion of alloy 1. Figure 2b reveals that the number fraction of LAGB in alloy 2 further increases by more than 7.5 times over alloy 1, and 47.2% grain boundaries are HAGB. By adding Sc to alloy 2, the average grain boundary angle is reduced to 21.70° , and the fraction of HAGB is reduced by 46.6%. It is suggested that the deformed structure and substructure with LAGB are retained in alloy 2 (Figure 1d), which is beneficial to the improvement in tensile properties.

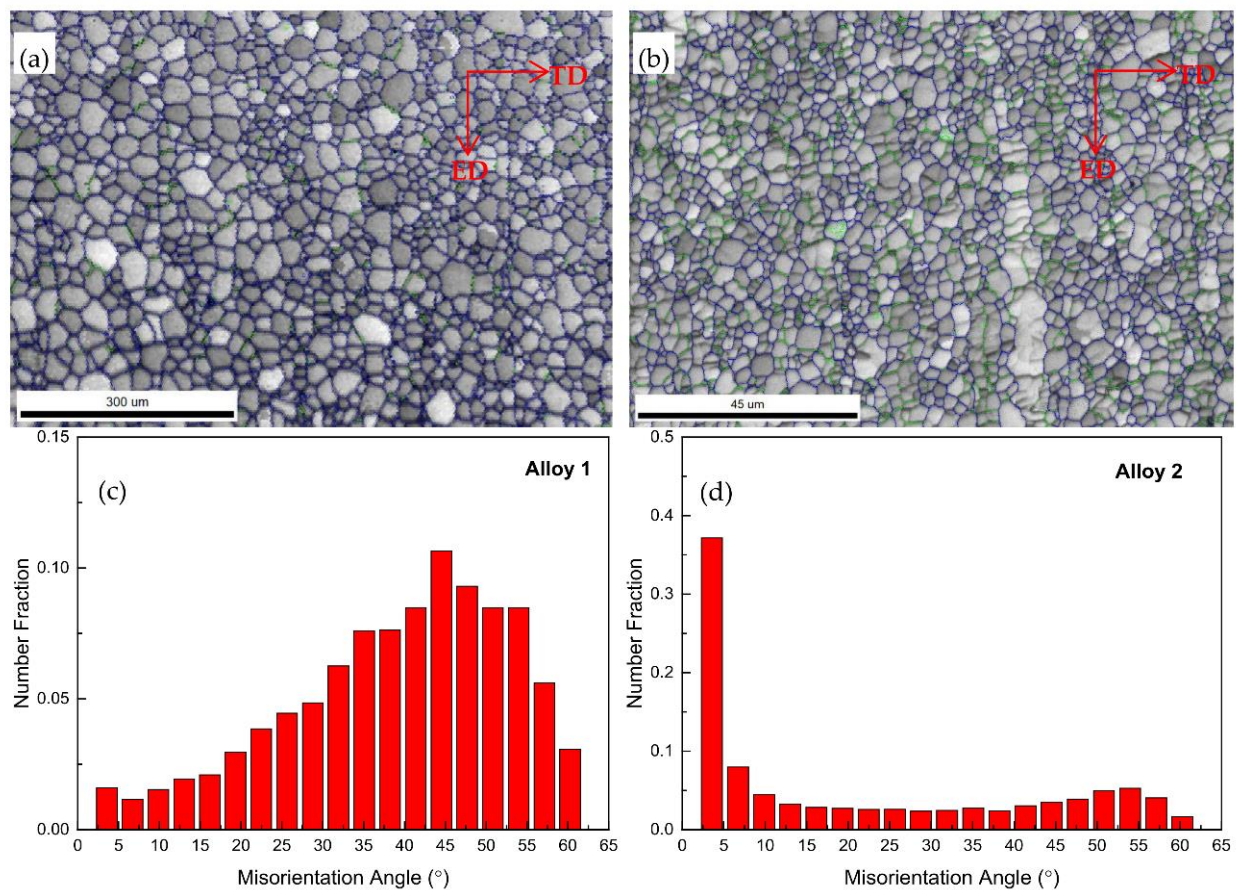


Figure 2. Grain boundary misorientation of alloy 1 (a,c) and alloy 2 (b,d) after hot extrusion: (a,b) GBM maps; (c,d) misorientation angle distribution.

Table 3. Comparison of statistical grain boundary misorientation for two categories of extruded Al-5Mg alloys.

	High Angle Grain Boundary		Low Angle Grain Boundary		Average Grain Boundary Angle
	f_{HAGB}	$\bar{\theta}_{HAGB}/^\circ$	f_{LAGB}	$\bar{\theta}_{LAGB}/^\circ$	$\bar{\theta}/^\circ$
Alloy 1	0.938	41.09	0.062	8.66	39.07
Alloy 2	0.472	40.19	0.528	5.17	21.70

Previous research has shown that Al-Mg alloy is prone to dynamic recrystallization (DRX) during hot deformation, which is a kind of high-level stacking fault energy metal [20]. In the early stage of hot extrusion, a large number of LAGBs (green lines in Figure 2b) are formed with severe stress concentration, accompanied by the evolution of substructures (yellow grains shown in Figure 1c,d). Then, the rate of partial dislocation formation increases on the LAGBs, resulting in an increase in the misorientation of the LAGB, which is converted into a HAGB (blue lines in Figure 2b). Finally, dynamic recrystallization is completed, and new recrystallized grains are formed, red grains shown in Figure 1c,d. There exist 74.3% recrystallized grains in alloy 1, indicating that maturely complete dynamic recrystallization occurred in alloy 1 (Figure 1c). The addition of Sc in alloy 2 inhibits recovery and recrystallization during hot extrusion due to the pinning of grain boundaries by Al_3Sc particles, resulting in a banding structure composed of a deformed structure and a substructure (Figure 1d) [13].

In the above, alloy 2 containing Sc is partially recrystallized during hot extrusion, whose uneven fine grains have a great effect on the mechanical properties of as-extruded Al-Mg alloy. The high angle grain boundary strengthening (σ_{HAGB}) caused by the presence of HAGB in the alloy can be directly calculated by the Hall-Petch formula (Formula (1)) [21]. While the low angle grain boundary strengthening (σ_{LAGB}) caused by the LAGB can be calculated by Formula (2). σ_{HAGB} and σ_{LAGB} are described by Formulas (1) and (2) as follows [22]:

$$\sigma_{\text{HAGB}} = k_{\text{H-P}} \left(f_{\text{HAGB}} / \bar{d} \right)^{1/2} \quad (1)$$

$$\sigma_{\text{LAGB}} = M\alpha G \left(3bf_{\text{LAGB}}\bar{\theta}_{\text{LAGB}} / \bar{d} \right)^{1/2} \quad (2)$$

where $k_{\text{H-P}} = 0.17 \text{ MPa} \cdot \text{m}^{1/2}$ is the Hall-Petch coefficient. f_{HAGB} , f_{LAGB} , $\bar{\theta}_{\text{LAGB}}$, and $\bar{d}$ are HAGB fraction, LAGB fraction, average LAGB misorientation angle, and average grain size, respectively, and the values are shown in Table 3. M and α are Taylor orientation factors, G is the shear modulus, and b is the Burgers vector whose values are shown in Table 4. As can be seen in Table 5, the sum of the yield strength increments due to grain boundary strengthening (σ_{GB}) in alloy 2 is 103.59 MPa, which is much higher than that of alloy 1. σ_{LAGB} is increased by a factor of 4.5, and σ_{HAGB} is increased by 73.4% compared to that of alloy 1. It is demonstrated that Sc has an excellent anti-recrystallization effect in Al-5Mg alloy during extrusion.

Table 4. Coefficients in the strength calculation formula [23–25].

Parameter	b/nm	λ/nm	M	α	G/GPa	$k_{\text{H-P}}/\text{MPa} \cdot \text{m}^{1/2}$
Value	0.286	0.154056	3.06	0.24	26	0.17

Note: b is the Burgers vector, λ is the wavelength of Cu-K α ray, M and α are Taylor orientation factors, G is shear modulus, and $k_{\text{H-P}}$ is the Hall-Petch coefficient.

Table 5. Dislocation strengthening and grain-boundaries strengthening of as-extruded Al-5Mg alloys.

	$\sigma_{\text{LAGB}}/\text{MPa}$	$\sigma_{\text{HAGB}}/\text{MPa}$
Alloy 1	9.60	29.22
Alloy 2	52.91	50.68

3.2. XRD Analysis and Dislocation Strengthening

Figure 3 displays the XRD patterns and full width at half maxima (FWHM) of as-extruded Al-5Mg alloys. As shown in Figure 3c, a major increment in the intensity of the (111) fundamental peak compared to that of the other peaks is observed, which may indicate texturing of the microstructure along the (111) plane. FWHM of alloy 2 is wider than that of alloy 1 in Figure 3b,d, and it shows that the lattice strain and dislocation density are increased in alloy 2.

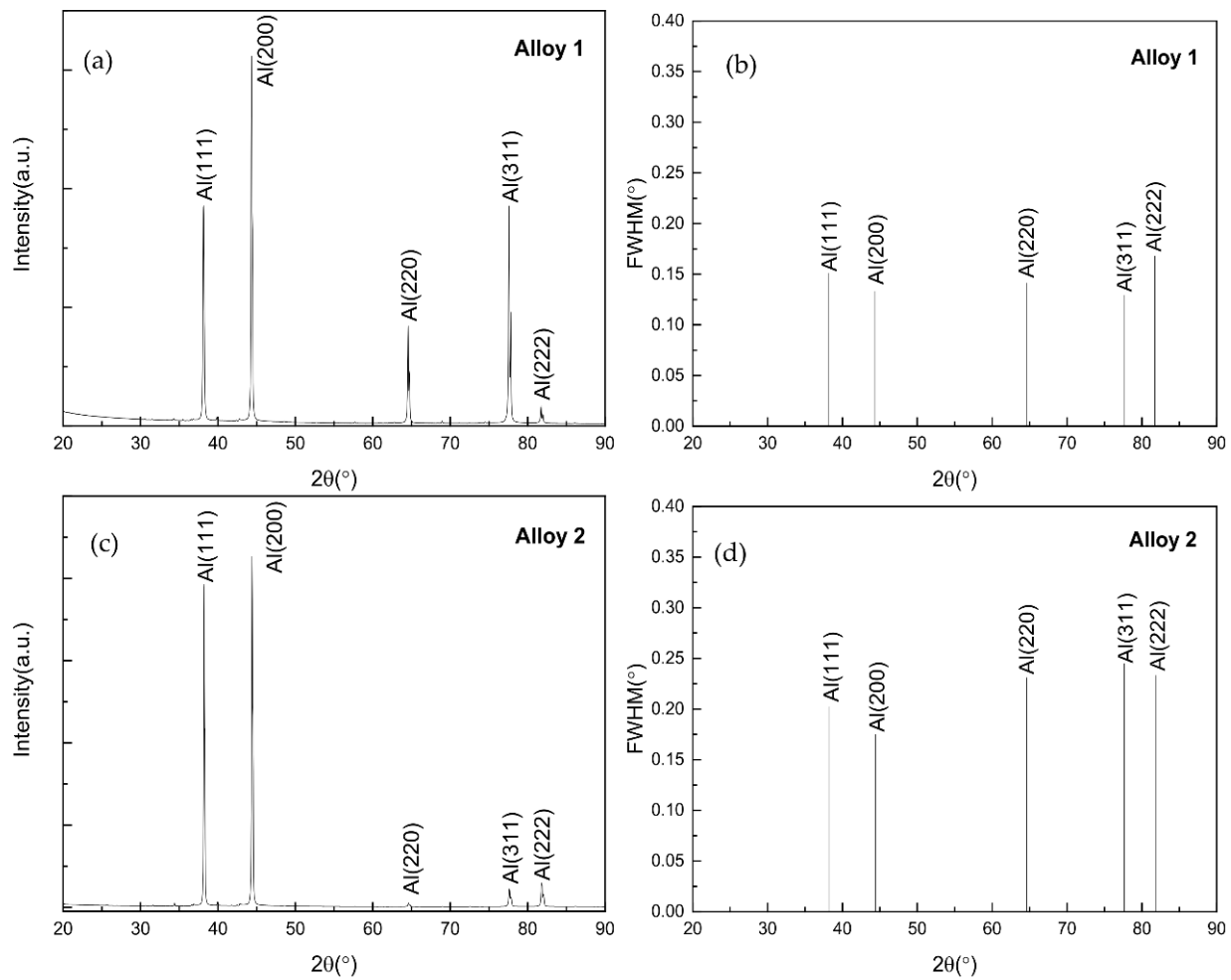


Figure 3. XRD analysis of alloy 1 (a,b) and alloy 2 (c,d) after hot extrusion: (a,c) XRD patterns; (b,d) FWHM.

The integral breadth analysis was used to calculate the coherent diffraction zone size and lattice strain from the XRD line broadening, leading to the following function [26]:

$$\frac{(\delta_{2\theta})^2}{\tan^2\theta_0} = \frac{\lambda}{d} \left(\frac{\delta_{2\theta}}{\tan\theta_0 \sin\theta_0} \right) + 25\langle e \rangle^2 \quad (3)$$

where $\delta_{2\theta}$ is the measured FWHM, θ_0 is the peak maximum position, λ is the wavelength of Cu-K α ray, d is the size of XRD coherent diffraction zone, and $\langle e \rangle$ is the lattice strain. Figure 4 shows the least-squares fit of the $(\delta_{2\theta})^2/\tan^2\theta_0$ against $\delta_{2\theta}/\tan\theta_0 \sin\theta_0$ for all measured peaks of as-extruded Al–5Mg alloys. The slope is λ/d and the intercept is $25\langle e \rangle^2$. The size of the XRD coherent diffraction zone (d) and the root mean square lattice strain ($\langle e^2 \rangle^{1/2}$) were calculated, and the results are listed in Table 6.

Table 6. Microstructural and mechanical features calculated from XRD.

	d/nm	$\langle e^2 \rangle^{1/2}$	ρ/m^{-2}	σ_{dis}/MPa
Alloy 1	73.01	1.459×10^{-4}	0.242×10^{14}	26.86
Alloy 2	49.97	2.435×10^{-4}	0.590×10^{14}	41.95

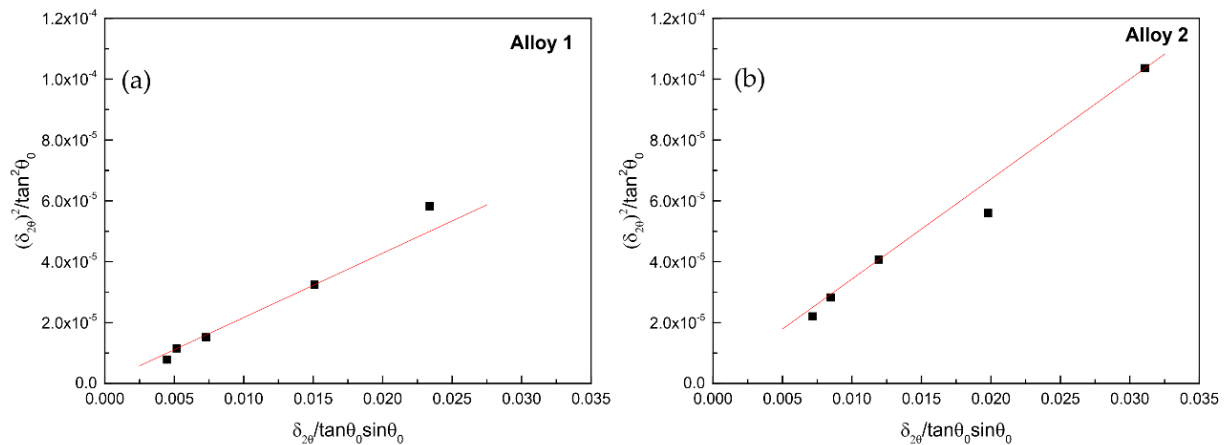


Figure 4. Integral breadth analysis to calculate the coherent diffraction zone size and lattice strain from XRD data.

The relationship between dislocation density (ρ), coherent diffraction zone size (d), and square root of lattice strain ($\langle e^2 \rangle^{1/2}$) can be described by the following Equation [27]:

$$\rho = 2\sqrt{3}\langle e^2 \rangle^{1/2} / (d \times b) \quad (4)$$

where b is the Burger vector, and the value of b is 0.286 nm [25]. The calculated dislocation density (ρ) values are listed in Table 4.

The contribution of dislocation strength (σ_{dis}) to yield strength (σ_y) can be calculated from Equation (3) [28]:

$$\sigma_{dis} = M\alpha Gb\rho^{1/2} \quad (5)$$

where M and α are Taylor orientation factors, G is the shear modulus, and b is the Burgers vector. The values of these parameters are shown in Table 4. The contribution of dislocation strengthening to yield strength in alloy 2 reaches 41.95 MPa, which is 15.09 MPa more than in alloy 1. It shows that the microalloying element Sc is beneficial in increasing the dislocation density of Al-5Mg alloy during extrusion deformation [29]. At the same time, the Al_3Sc second phase particles can effectively pin the dislocations and further improve the strength of the Al-Mg alloy [30].

3.3. Mechanical Property

Figure 5 depicts the room temperature tensile mechanical properties of as-extruded Al-5Mg alloys. It can be seen that the tensile strength (maximum resistance (R_m)) of alloy 2 is 380.7 MPa, which is 34.1% higher than that of alloy 1 without the Sc element. Meanwhile, the proof strength of non-proportional (proof resistance ($R_{p0.2}$)) increased by 93.0% over alloy 1. However, alloy 2 has a 46.2% reduction in elongation (A) compared to alloy 1.

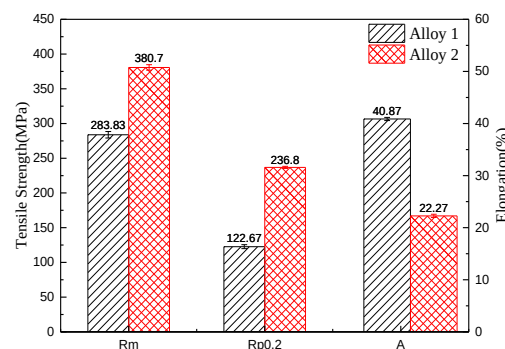


Figure 5. The mechanical properties of the extruded Al-5Mg alloys.

Figure 6 illustrates the tensile fracture morphology of the extruded Al-5Mg alloys. The macroscopic morphologies of the tensile fracture of the two alloys show high ductility with typical cup-cone surface morphology in Figure 6a,d, in which the shear lip region in the fracture of alloy 1 is wider than that of alloy 2, resulting in better fracture toughness of alloy 1 [31]. It is found that the dimples of alloy 1 are deeper, and the dimple size is larger, as shown in Figure 6b,c. While the dimples of alloy 2 are shallower, the dimple size is smaller, and the number of dimples is larger in Figure 6e,f. It is inferred that alloy 2 has higher strength and lower elongation compared to alloy 1.

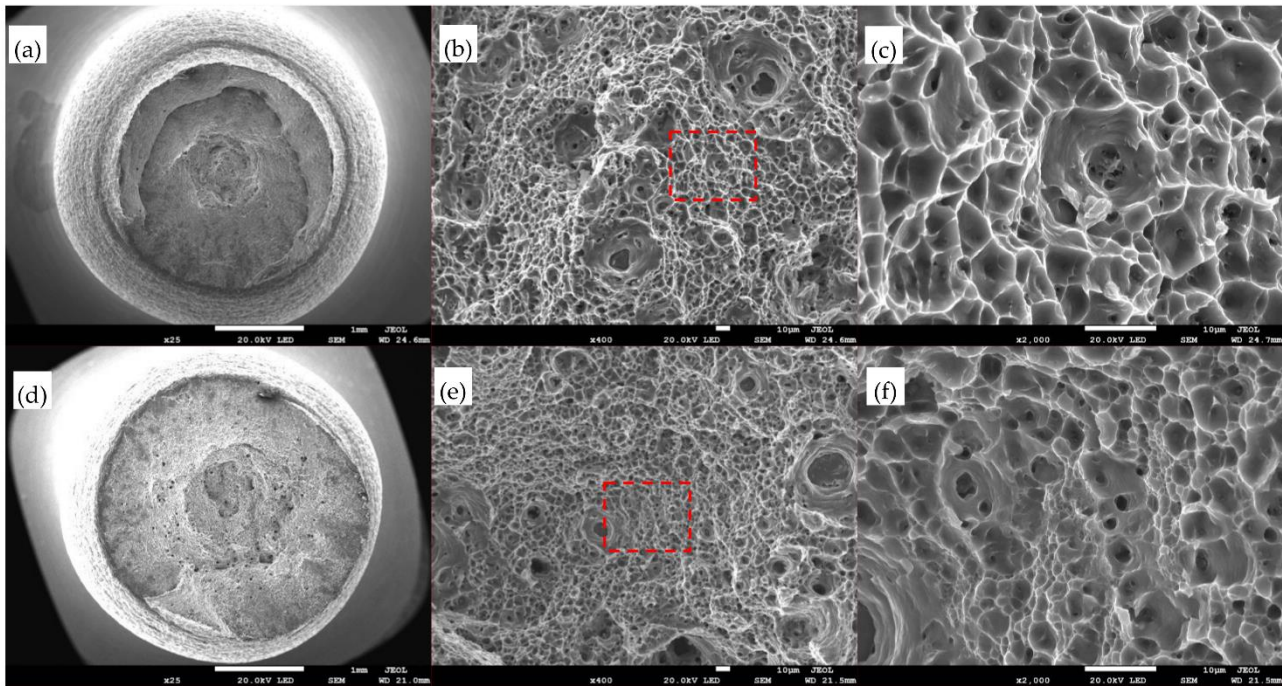


Figure 6. SEM micrographs of tensile fracture morphology of alloy 1 (a–c) and alloy 2 (b–f) after hot extrusion: (a,d) macro fracture; (b,e) high magnification; (c,f) high magnification of the dashed parts of (b,e).

According to the microstructure characteristics of alloy 2 in Figure 1b,d, it can be confirmed that the average grain size of alloy 2 is obviously refined, and a partially narrow banding structure is preserved, which leads to an increase in the hindering effect of grain boundaries on dislocation movement and further increases the strength [32]. In addition, the existence of fine Al_3Sc particles in alloy 2 not only hinders dislocations from improving the strength but also causes stress concentration, resulting in a decrease in the elongation of alloy 2 [33].

4. Conclusions

In this research, two categories of Al-5Mg alloys were fabricated by hot extrusion process after vacuum + argon-protected casting. The effects of Sc microalloying on microstructure and properties of as-extruded Al-5Mg alloy were investigated. The following conclusions can be made:

- (1) Partial recrystallization was observed in alloy 2 with 0.2% Sc content, resulting in a structure consisting of 21.8% equiaxed recrystallized structure and 78.2% fine banding structure. The grain size of alloy 2 is about $5.31\ \mu\text{m}$, which is 83.3% finer than that of alloy 1 without Sc;
- (2) The addition of Sc leads to an increase in grain boundary strengthening and dislocation strengthening in alloy 2, reaching 103.59 MPa and 41.85 MPa, respectively;

- (3) Alloy 2 with Sc has a higher tensile strength of 380.7 MPa, which is 34.1% higher than that of alloy 1, but alloy 2 has a 46.2% reduction in elongation compared to alloy 1.

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