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Cast Microstructure and Crystallographic Features of Al₃Sc Dendrites in High Sc-Contained Al-Sc Alloys

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Abstract: Al-Sc alloys containing high Sc content are employed as sputtering targets for the fabrication of high-performance piezoelectric films during magnetic sputtering. Due to the high proportion of the Al₃Sc phase, their workability is quite limited, and they are often used in the as-cast state. In this study, the crystallography of Al₃Sc dendrites in as-casted Al-10at.%Sc and Al-20at.%Sc samples is examined using electron backscatter diffraction (EBSD). With increasing Sc content, the fraction of Al₃Sc also increases. The Al₃Sc dendrites exhibit a cubic relationship with the Al matrix in both alloys. However, in Al-10%Sc alloys, the facets of the Al₃Sc dendrites are parallel to {001} planes, while twinning is observed in Al-20at.%. The twinning plane is parallel to the {111} plane, and the dendrite growth direction aligns with the <110> directions. The different morphologies of the dendrite structures in these two alloys are discussed in relation to thermodynamic and kinetic considerations based on the phase diagram and nucleation rate.

Keywords: Al-Sc alloy; Al₃Sc; twin; dendrite; crystallography



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1. Introduction

Scandium (Sc) is often doped into AlN (Al_{1-x}Sc_xN) to enhance its piezoelectric properties [1–5]. The effectiveness of this enhancement increases with higher Sc content, and the content of Sc can be up to 40 at.% [1,6], below which the hexagonal structure of Al_{1-x}Sc_xN can be maintained. Otherwise, different properties would result from a different crystal structure.

The Al_{1-x}Sc_xN films could be produced by reactive magnetic sputtering [7–9], a method highly attractive for mass production. The Al-Sc alloy target plays a crucial role in the sputtering process, and the specific Al-Sc alloy target is sputtered and combined with nitrogen to form the Al_{1-x}Sc_xN film. The doping content of Sc in the film can be controlled by using different Al-Sc alloy targets with varying compositions. Consequently, the quality of Al_{1-x}Sc_xN films depends on the properties of the Al-Sc alloy targets, such as their chemical composition, chemical homogeneity, grain size, texture, etc. [5]. According to the Al-Sc binary phase diagram [10], Al₃Sc will be formed when the Sc content exceeds 0.55 wt.% (0.33 at.%) and its fraction increases with higher Sc content. Therefore, understanding the microstructure in as-casted Al-Sc alloys, especially for high Sc content, becomes important.

In an aluminum alloy, a small amount of Sc is often applied to refine grain size [11,12]. During the casting process, the Al₃Sc compounds with a L1₂ (Cu₃Au) type structure form initially, and subsequently, aluminum grains inhomogeneously nucleate on the Al₃Sc compounds. However, previous work focuses on conventional aluminum alloys for structural applications with Sc content typically lower than 1 wt.% [13–17]. The variation in the morphology of Al₃Sc particles from cuboidal to dendritic and seaweed-like shapes with an

increase in the cooling rate indicates the influence of cooling conditions on the solidification process [18,19]. Different cooling rates can lead to variations in the nucleation and growth of phases during the solidification of the alloy [20]. The specific shapes observed can be attributed to the intricate interplay of factors such as nucleation kinetics, crystallography, and thermodynamics under varying cooling rates. This information is crucial for understanding the microstructural evolution and properties of the alloy under different processing conditions.

The limited study on the Al-Sc alloy with higher Sc content (such as more than 10 at.% or 15 wt.%) is attributed, in part, to cost considerations. Higher scandium content can significantly increase the expense of alloy production. As a result, investigations on Al-Sc alloys with higher Sc content are rarely reported in the literature [21]. However, this type of alloy is of great interest to the industry due to its significance in producing $\text{Al}_{1-x}\text{Sc}_x\text{N}$ films. In the Al-Sc alloy with high Sc content, the fraction of the Al_3Sc phase may be dominant [10], and the Al-Sc alloy target can be used only in the as-casted state. This is because the Al_3Sc compounds are difficult to deform [22] and can lead to cracking in practical applications.

Therefore, this study will investigate the formation mechanism of the as-casted Al-Sc alloys with high Sc content, providing valuable insights into the microstructure and crystallography and offering guidance for materials processing and target fabrication.

2. Materials and Methods

In this study, Al-10%at.Sc and Al-20%at.Sc alloys, denoted as Al-10Sc and Al-20Sc for simplification, were investigated. High-purity aluminum with a purity of ≥ 99.9995 wt.% is used as the base metal, and high-purity scandium with purity of ≥ 99.99 wt% is added. Alloys with nominal composition of Al-10%at.Sc and Al-20%at.Sc alloys were cast using a graphite mold. The composition in different parts of our samples was examined by ICP-OES, and the variation of Sc content was found to be less than 4.6%. Since high-purity aluminum and scandium are used, the presence of other elements is extremely low, which is critical for preparing high-quality films by sputtering method. The casted samples underwent mechanical grinding, polishing, and additional etching using a hydrofluoric acid (HF) solution. Microstructural analysis was conducted using optical microscopy (ZEISS Axio Lab.A1, Zeiss, Oberkochen, Germany), scanning electron microscopy (ZEISS Gemini 2, Zeiss, Oberkochen, Germany) equipped with an electron backscatter diffraction (EBSD) system (Oxford Symmetry 2, Oxford instruments, Abingdon, England), and transmission electron microscopy (TEM, JEM200, JEOL, Tokyo, Japan). Electrolytic polishing was conducted using an electrolyte composed of high chloric acid and ethanol, with a volume ratio of $V(\text{high chloric acid}):V(\text{ethanol}) = 1:9$, and a voltage of 25~30 V. Conventional EBSD data analysis, including pole figure, grain types, etc., were processed using AZtec Crystal software (Version 2.1, Oxford instruments, Abingdon, England) and the open-source software for transformation crystallography PTCLab (Version 1.5, open software, Beijing, China) [23]. The orientation relationship between Al and Al_3Sc is characterized by orientation mapping. Furthermore, the growth direction of the Al_3Sc dendrites is analyzed through trace analysis based on the statistical EBSD data [24,25].

3. Results

3.1. Microstructure

Figure 1a,b shows the optical microstructure of Al-10Sc and Al-20Sc alloy, respectively. The dark grey contrast in both images is Al_3Sc , and the light grey color is an Al matrix. Due to the increase in Sc content, the alloys are dominant with Al_3Sc . According to the Al-Sc binary phase diagram [10], a eutectic transformation from L (liquid) to $\alpha\text{-Al} + \text{Al}_3\text{Sc}$ occurs at approximately 665 °C, with a eutectic composition of 0.55 wt.%. The equilibrium phase consists of primary Al_3Sc and eutectic structures. The fraction of the Al_3Sc for the Al-Sc alloy is 43.9% and 88.1%, compared to 40% and 80% for equilibrium calculation based on the phase diagram [10]. The observed fraction is larger than the equilibrium value, which

may be due to the non-equilibrium effect during the casting process. In addition, the size of the Al_3Sc particle is about $13.3\ \mu\text{m}$ in the Al-10Sc alloy.

Notably, in Figure 1, the regular shape of Al_3Sc with well-defined facets is found in the Al-10Sc alloy, while irregular dendrite is found in the Al-20Sc alloy. It seems the Sc content will affect the morphologies of Al_3Sc compounds. The crystallography of Al_3Sc compounds will be further determined using electron backscatter diffraction (EBSD).

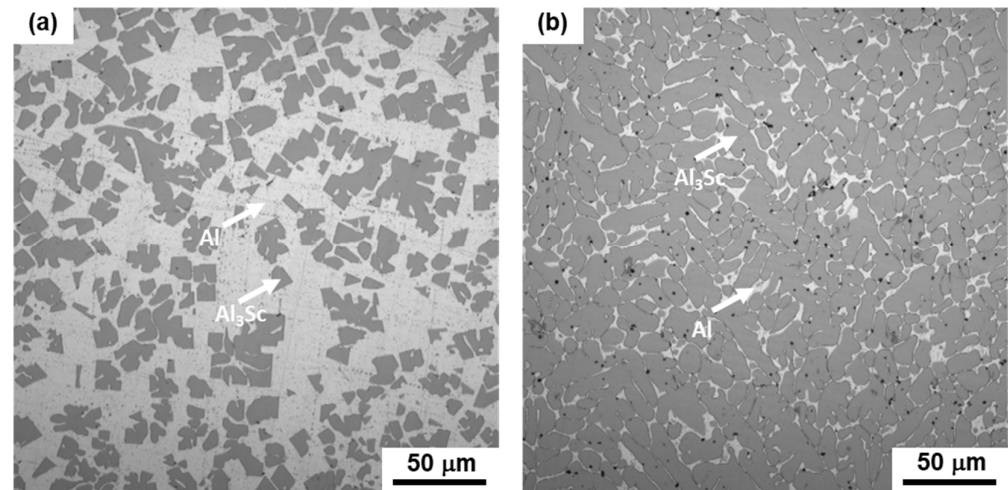


Figure 1. Optical microstructure for the following alloys: (a) Al-10Sc; (b) Al-20Sc.

3.2. EBSD Results

Figure 2a,b shows the orientation mapping for the Al-10Sc alloy and the Al-20Sc alloy, respectively. Different colors in these mappings show different orientations, and the shape of the grain can be visualized. In general, the same color indicates the same orientation and one grain. The grains are nearly equiaxed in Al-10Sc and elongated in Al-20Sc, where planar twin boundary interfaces can be observed, which will be determined later. Note that the Al_3Sc phase cannot be resolvable in the EBSD mapping in Figure 2 since they show the same color as the neighboring Al matrix. This is because of similar lattice parameters and structures between the Al and Al_3Sc phases and a cubic orientation exhibited between the two phases

$$\langle 100 \rangle_{\text{Al}} // \langle 100 \rangle_{\text{Al}_3\text{Sc}}$$

$$\langle 001 \rangle_{\text{Al}} // \langle 001 \rangle_{\text{Al}_3\text{Sc}}$$

as often reported in previous studies [11,12].

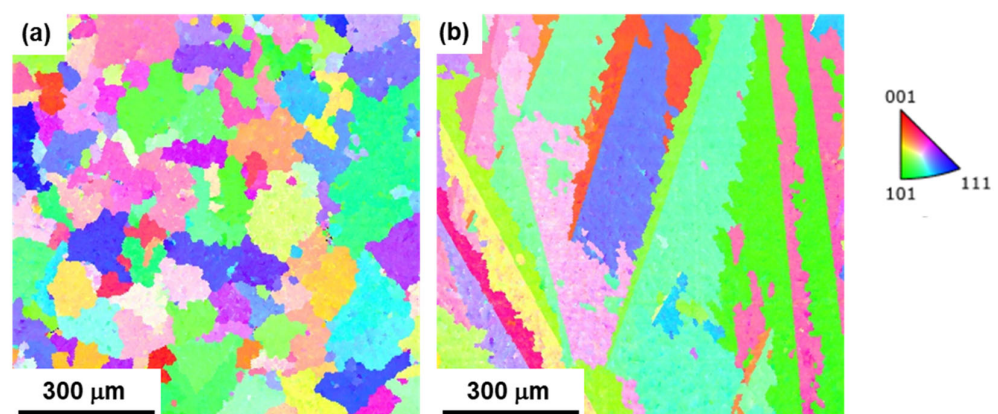


Figure 2. Orientation mapping for the following alloys: (a) Al-10Sc; (b) Al-20Sc. The color corresponds to the normal direction as indexed according to the inverse pole figure displayed on the right.

To confirm the result, energy dispersive X-ray spectroscopy (EDS) was applied together with the EBSD method to identify Al and Al_3Sc since the EBSD method alone made it hard to differentiate between two phases due to similar lattice parameters and structures. An EBSD result observed from Al-20Sc is shown in Figure 3. Figure 3a shows the orientation mapping, and Figure 3b is the corresponding phase mapping, where Al is distributed between the Al_3Sc dendrites. The green color in the phase mapping is Al_3Sc , and the red color is for Al. Clearly, Al and Al_3Sc can be differentiated by combining chemical composition analysis with the EBSD method. In the orientation mapping, Al shows the same orientation color as neighboring Al_3Sc , confirming a cubic relationship between the two phases. Additionally, the EBSD method can reveal detailed structures, such as the planar interface between dendrites in Figure 3a.

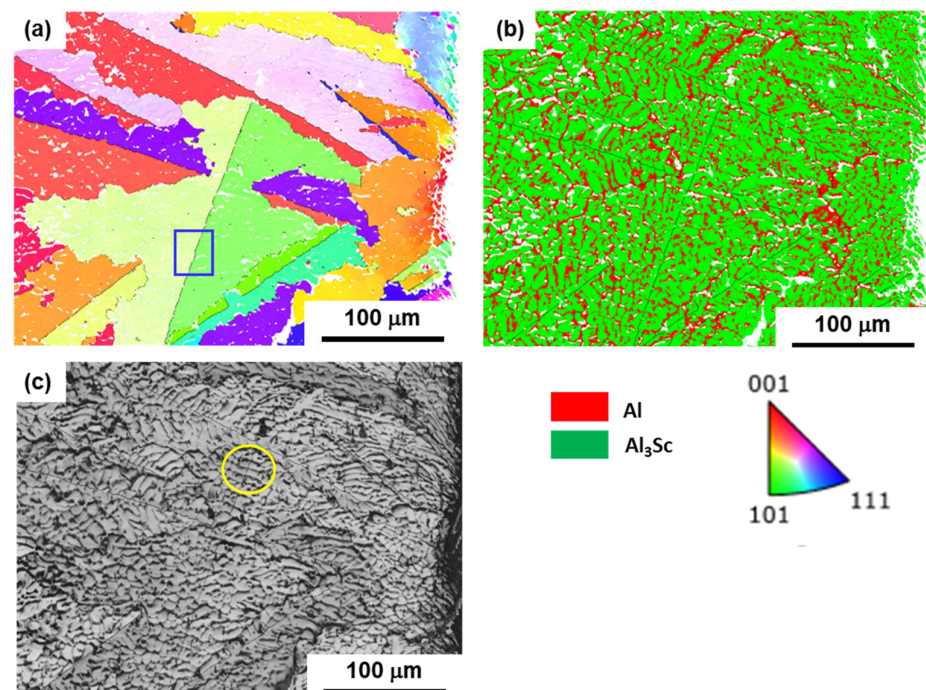


Figure 3. EBSD results for Al-20Sc alloy: (a) Orientation mapping; (b) Phase mapping; (c) Band contrast.

The pole figures can be used to analyze the orientation relationship visually. The $\{001\}$, $\{011\}$, and $\{111\}$ pole figures for the phases around the flat interface marked by a rectangle in Figure 3a are shown in Figure 4a–c. According to the pole figures, they show a twin relationship, and they are rotating 60° degrees relative to each other around a $\langle 111 \rangle$ direction, marked with a triangle in Figure 4a,b. The common rotation axis is further confirmed in Figure 4c, where a pair of $\langle 111 \rangle$ poles coincide with each other in the $\{111\}$ pole figure. Similar analyses have been carried out to confirm the twin relationship, and the twin structures can be formed during the casting process. Apart from the orientation relationship, the flat interface is analyzed with trace analysis shown in Figure 5.

In Figure 5, the flat interface traces observed in the EBSD data are segmented and plotted in the stereographic projection with red dots, along with their crystallographically equivalent directions by symmetry operations. The red dots in Figure 5 are the projections of the trace directions. The patterns of the dots resemble the symmetry of the crystal symmetry. Remarkably, the projection traces are closely situated on the big circles corresponding to $\{111\}$ planes, indicating that the interface traces lie on the $\{111\}$ planes. Therefore, the flat boundary is parallel to $\{111\}$ planes, which are characteristic coherent twin boundaries in face-centered cubic structures. Note that the $\{111\}$ twin is most likely for Al_3Sc phases according to the orientation and phase mapping in Figure 3. Due to the low energy nature, these twin boundaries are challenging to observe in optical microscopy but become visible

through orientation mapping, as shown in Figure 3. Moreover, the traces are clustering along the $\langle 011 \rangle$ poles, indicating twin growth direction growth along the $\langle 011 \rangle$ directions. Since the interface shows well-defined facets and the interface runs along low-indexed planes in the present study, the trace analysis method applied in the literature [24,25] is not shown here.

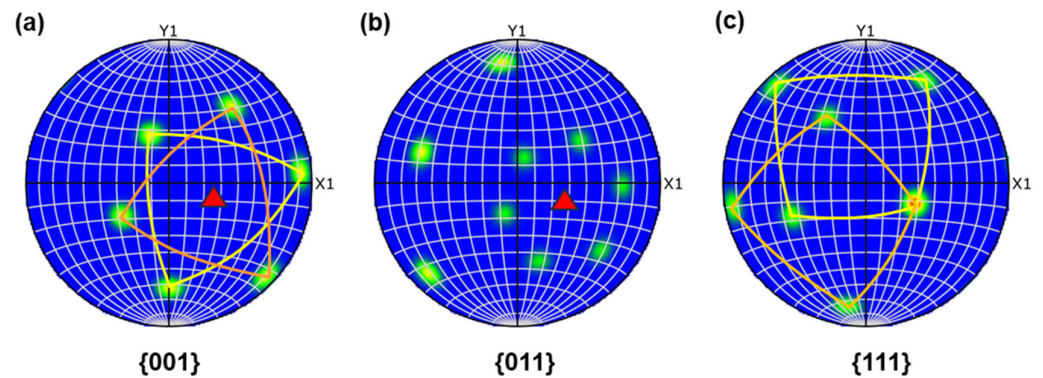


Figure 4. Pole figures showing the orientation around the boundary marked by a rectangle in Figure 3a: (a) {001}; (b) {011}; (c) {111}.

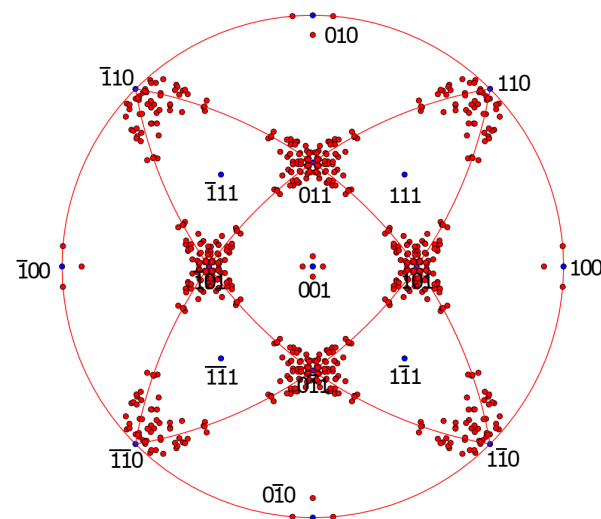


Figure 5. The stereographic projection of traces for twin boundaries, where the red dots are the directions of traces.

3.3. Dendrite Crystallography

Based on the EBSD mapping, a cubic orientation relationship between the Al_3Sc and Al is found. It is also observed that the Al_3Sc in Figure 1a for Al-10Sc alloy exhibits flat and faceted interfaces. Next, the interfaces between the two phases are analyzed by trace analysis similar to Figure 5. The results are presented in Figure 6a, where the traces are located near $\{001\}$ big circles. Therefore, the interface between Al and Al_3Sc is parallel to $\{001\}_{\text{Al}}$ and $\{001\}_{\text{Al}_3\text{Sc}}$.

Furthermore, the dendrite directions for Al_3Sc in the Al-20Sc alloy are shown in Figure 6b. The directions generally lie in the $\{001\}$ planes and are close to the $\langle 110 \rangle$ or $\langle uv0 \rangle$ directions. The projection is a result of the sectioning of the dendrite from three-dimensional space, and deviations from the actual dendrite direction may be observed. However, based on the statistical data from EBSD, the trends for such directions can be captured. In this case, the dendrite grows along the $\langle 011 \rangle$ directions.

4. Discussion

4.1. Microstructure Variation

The two alloys investigated in this paper contain 10 at.% Sc and 20 at.% Sc. During solidification, Al_3Sc is formed first, establishing a phase equilibrium between the liquid phase and Al_3Sc . According to the thermodynamic relationship between the liquid phase and the compound [26], the solubility (x) of the compound in the liquid generally follows an exponential relationship with temperature (T):

$$x = ke^{-\frac{Q}{RT}} \quad (1)$$

where k is the coefficient, R is the gas constant, and Q is the enthalpy. After taking the logarithm of both sides of the equation, the relationship between $\ln x$ and $1/T$ can be expressed as follows:

$$\ln x = \ln k - \frac{Q}{RT} \quad (2)$$

According to the Al-Sc phase diagram [10], the solubility curve between the liquid phase and Al_3Sc was sampled from the phase diagram and fitted based on Equation (2), and the results are shown in Figure 8. The slope of the straight line is -7860.3 , yielding $Q = 65.32 \text{ kJ/mol}$, and the intercept is 7.95 , indicating k is 2.8×10^3 . The actual solid solubility (solid circles in Figure 8) aligns well with the fitted curve. The solubility of Al_3Sc in Al is given by

$$x\% = 2.8 \times 10^3 e^{-\frac{7860.3}{T}} \quad (3)$$

According to Equation (3), the melting temperature can be calculated for high-Sc alloys with $x < 25\%$. For example, when $x = 10 \text{ at.}\%$, the melting temperature is $1122.0 \text{ }^\circ\text{C}$, and when $x = 20 \text{ at.}\%$, the melting temperature is $1317.6 \text{ }^\circ\text{C}$. In the following, the undercooling for different alloy compositions will be calculated based on the solidification temperature, discussing the solidification behavior of alloys with varying Sc compositions.

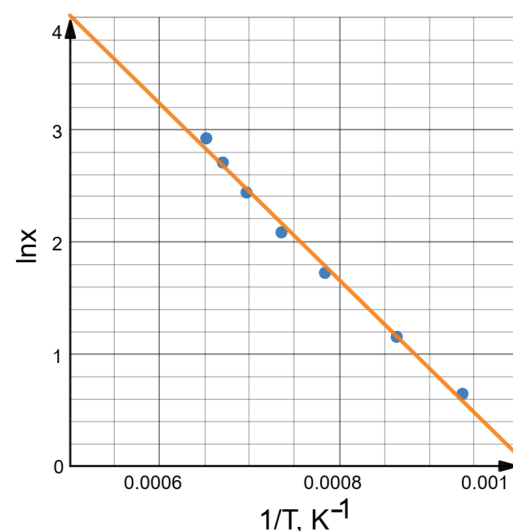


Figure 8. The solubility between the liquid phase and Al_3Sc phase was fitted from the phase diagram. The dots are adopted from the phase diagram, and the line is fitted result.

When the liquid phase solidifies, according to classical nucleation theory [26,27], the critical nucleation radius is given by the following:

$$r^* = \frac{2\gamma V_m}{\Delta G} \quad (4)$$

and the nucleation energy barrier is given by the following:

$$\Delta G^* = \frac{16\pi\gamma^3 V_m^2}{3\Delta G^2} \quad (5)$$

where γ is the interfacial energy, ΔG is the nucleation driving force (in J/mol), and V_m is the molar volume of Al_3Sc . The driving force for the formation of compounds during liquid phase solidification is as follows [1]:

$$\Delta G = \frac{Q}{4} \cdot \frac{T_m - T}{T_m} \quad (6)$$

where Q represents the value in Equation (1) or (3), T_m is the melting point of the given alloy (which can be calculated according to Equation (3) for a given alloy composition), and T is the temperature. As the Sc content increases, T_m also increases, leading to a larger driving force ΔG , as shown in Figure 9a. The difference in the driving force for Al-10Sc and Al-20Sc is about 600 J/mol. A larger driving force in Equation (5) results in a lower critical nucleation energy barrier, promoting nucleation. Taking the Al/ Al_3Sc interfacial energy as 0.23 J/m² [28], the critical nucleation radius for the 20% Sc alloy is smaller than that for the 10% Sc alloy at the same solidification temperature, as shown in Figure 9b. Additionally, the diffusion coefficient of Sc in the liquid phase is 10^{−9} m²/s [18]. Therefore, the nucleation rate of Al_3Sc can be determined, as shown in Figure 9b. Below a certain degree of undercooling, the nucleation rate of Al_3Sc sharply increases, and when the nucleation rate increases rapidly, the critical nucleus size is approximately 1 nm. Under the same degree of undercooling conditions, the nucleation rate of the high-Sc alloy is also higher than that of the low-Sc alloy samples. These results can also be extended to samples with different cooling rate. Nevertheless, the ease of nucleation at high temperatures leads to a higher growth rate of the sample, as evidenced by the larger dendrites in Figure 2b.

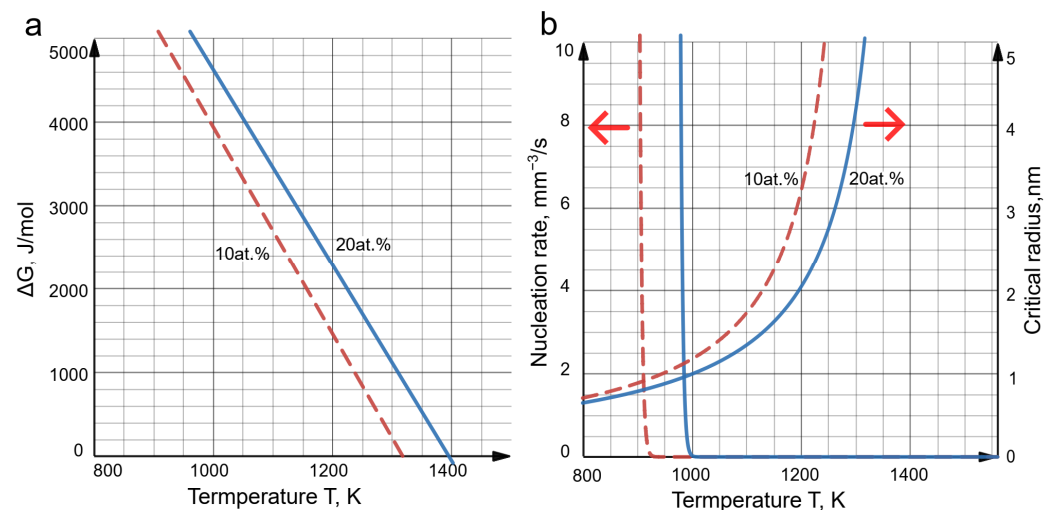


Figure 9. (a) Nucleation driving force. (b) Critical nucleation radius and nucleation rate. The red dot lines are for Al-10Sc alloy, while blue solid lines are for Al-20Sc alloy.

4.2. Morphology Difference

According to Jackson's interface roughness parameter α [26], when $\alpha > 2$, the interface is a smooth interface, where the interface is either fully occupied by atoms or entirely in the

liquid phase. When $\alpha \leq 2$, the interface is a rough interface. The roughness parameter α can be expressed as the following:

$$\alpha \approx \left(\frac{z^*}{z} \right) \frac{\Delta H_m}{RT_m} \quad (7)$$

z is the coordination number of the crystal, and z^* is the atomic coordination number on the exposed surface. For Al_3Sc with a simple cubic structure as for Al_3Sc , z is 6. Taking {001} as an example, $z^* = 4$. According to the data in Table 1 and Equation (3), it can be concluded that $\alpha \approx 3.4 > 2$. Therefore, a smooth interface is stable. The values of α for other interfaces are shown in Table 1. Since the interface energy of the {001} surface of the Al_3Sc compound is relatively low and the growth of smooth interface steps is slow [20], a cube consisting of {001} surfaces should form in equilibrium or during slow cooling, as reported in the literature [18,19]. This is also consistent with our 10% Sc alloy, which tends to form {001} planes. In the case of high-Sc alloys, the interface may become unstable due to the increased Sc content, leading to dendritic growth.

Table 1. Parameters for Al_3Sc Interface Roughness Calculation.

Melting point $T_{\text{Al}_3\text{Sc}}$	1593 K	[29]
Melting enthalpy ΔH	67,158 J/mol	[29]
Mole volume	$10.4 \times 10^{-6} \text{ m}^3/\text{mol}$	[30]
Coordinate atoms on {001}	4	
Coordinate atoms on {011}	2	
Coordinate atoms on {111}	0	
α for {001}	3.4	
α for {011}	1.7	
α for {111}	0	

During the solidification process of non-pure components, after the solidification of Al_3Sc , a Sc-depleted region is formed at the liquid front, leading to undercooling caused by different compositions, known as compositional undercooling. At this point, a planar interface becomes unstable. The conditions for the occurrence of compositional undercooling or interface instability are given by the following [20]:

$$\frac{m_L C_0}{D_L} \frac{1 - k_0}{k_0} \geq \frac{G_L}{v} \quad (8)$$

Here, k_0 is the solute partition coefficient, D_L is the liquid diffusion coefficient, m_L is the slope of the liquidus line, C_0 is the initial composition, G_L is the temperature gradient at the liquid front of the interface, and v is the solidification rate. A larger C_0 leads to a greater tendency for compositional undercooling, favoring the development of dendrites. Therefore, the alloy with 20% Sc exhibits a more developed dendritic structure compared to the alloy with 10% Sc. The quantitative comparison of the compositional undercooling is difficult without knowing the controlled solidification parameters. The origin of the twins also needs to be explored further.

4.3. The Crystallography of Al_3Sc

In either the Al-10Sc or Al-20Sc alloy, the cubic orientation relationship is held between Al and Al_3Sc compounds due to similar lattice parameters and small interphase misfit. As a result, the interfacial energy is low due to the misfit in the interface being less than 5% [31]. However, Al-10Sc, the low energy {001} facets are observed, and a cuboidal shape would be expected. The dendrite in this alloy, if there are any, grows along the $\langle 011 \rangle$ direction

with small $\{001\}$ facets, as shown in Figure 10a. However, the $\{111\}$ twinned dendrites are found in Al-20Sc in Figure 10b, where twin-oriented feathery grains are suggested to be new features together with columnar grains and equiaxed grains [32], where it originates from the stacking fault generated by the stress on the solidified part due to forced flow of liquids or convection [33,34]. The growth direction of twins is along the $\langle 011 \rangle$ directions, and the dendrite within the twins also grows near the $\langle 011 \rangle$ direction.

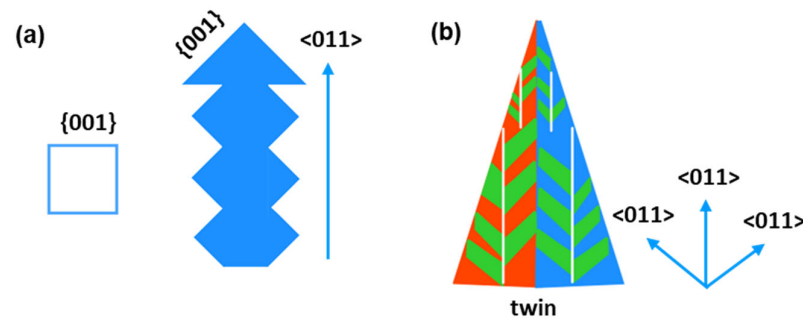


Figure 10. Schematic diagram for the crystallography for Al_3Sc in (a) Al-10Sc and (b) Al-20Sc.

As mentioned before, the dendrites originate from the phase instability due to the kinetic roughening from compositional supercooling. The morphologies of the Al_3Sc compound in Al-Sc alloys with Sc content below 1.5 wt.% show similar features [35]. However, the growth direction in cubic materials is usually along $\langle 100 \rangle$ [20], which is usually affected by thermal conductivity and elastic properties and external field including the temperature field, solute field, fluid field, etc. Henry et al. attributed these features to local solidification conditions [33]. The variation of the morphology and twin structure depends on various factors, and limited thermodynamic and kinetics factors have been considered. Further explorations into this aspect may be conducted.

5. Conclusions

The study investigates Al-Sc alloys with high Sc content (10% and 20% Sc) due to their importance in producing $\text{Al}_{1-x}\text{Sc}_x\text{N}$ films with Al-Sc alloy targets. The alloys are characterized by various microscopy techniques, and the present study focuses on understanding the microstructure and crystallography essential for materials processing and alloy target fabrication for high Sc-contained Al-Sc alloys.

- (1) With an increase in Sc content, the fraction of the Al_3Sc phase also increases, and this change in Sc composition affects the morphology of the alloy, from faceted cuboidal to twinned dendrites.
- (2) The Al_3Sc phases exhibit a cubic relationship with the Al matrix in both Al-10%Sc and Al-20%Sc alloys. The statistical analysis using EBSD data reveals that in Al-10%Sc alloys, the facet of the Al_3Sc compound is parallel to the $\{001\}$ plane. In contrast, a twin dendrite structure is observed in the Al-20%Sc alloy, with the twinning plane parallel to the $\{111\}$ plane and dendrite growth occurring along the $\langle 110 \rangle$ directions.
- (3) The coarsen Al_3Sc observed in the 20%Sc alloy is attributed to nucleation and a fast growth rate at high temperatures. The dendrite formation originates from interface instability and prefers growth along the $\langle 110 \rangle$ directions. The dendritic shape is more pronounced in Al-Sc alloys with higher Sc content. The twin structure is easily observed in the dendrites in Al_3Sc structures.

Author Contributions: J.H. contributed to the investigation and draft preparation. Q.J. contributed to the casted alloy preparation and data analysis. X.W., X.C. and Z.C. contributed to data curation and visualization. Z.D. contributed to conceptualization and analyzation. X.G. contributed to conceptualization, supervision, and data analysis. All authors were involved in writing and reviewing the manuscript. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: Authors: He, J.; Jia, Q.; Ding, Z.; Wang, X.; Cao, X.; Cao Z. were employed by the company GRIKIN Advanced Material Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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