**Effect of Si Content on the Morphology Evolution of the Si Primary Dendrites in Al-Si Alloy Solvent Refining Process**

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**Abstract**
Solvent refining with Al-Si alloy is a promising purification method for the production of solar-grade silicon (SoG-Si) feedstock owing to the advantages of low production cost and high impurity removal efficiency. In this process, larger refined Si primary dendrites should be easily collected after acid leaching, which is favorable to recovery, thereby reducing the production cost. Hence, the growth behavior of the precipitated Si crystal must be investigated systematically. In the present work, the morphology evolution of solidified Al-Si alloys with a wide range of Si content (30 ~ 70 wt%) was analyzed. The typical plate-like Si primary dendrites grown following the twin plane re-entrance edge (TPRE) mechanism formed in all alloy compositions. As increasing the Si content from 30 wt% to 50 wt%, the Si primary dendrites underwent a coarsening process attributed to the preferred growth along with < 211 > and < 111 > directions, leading to an increase in the experimental recovery rate. However, the preferred growth along < 211 > direction was inhibited when the Si content is higher than 55 wt%. Moreover, the broken effect originating from grain collision and thermal stress on the Si primary dendrites was enhanced by further increasing the Si content, resulting in a decrease in the experimental recovery rate. Therefore, the optimum composition is determined as Al-50 ~ 55 wt% Si for solvent refining solution, based on the cost reduction consideration.

**Keywords** Solvent refining · Al-Si alloy · Si primary dendrites · Recovery rate

**1 Introduction**
Solar cell production based on Si wafers has increased significantly over the decades, as there is a growing demand for clean energy. Material resources for this application are mainly high-purity solar-grade silicon (SoG-Si), either for single crystal wafers or for multi-crystalline wafers. Currently, the SoG-Si feedstock is mainly produced via the traditional Siemens process or its modified alternatives [1], which is fairly energy-intensive and environment-unfriendly [2]. As an alternative, the metallurgical refining processes, which employ metallurgical grade Si (MG-Si) as the starting materials, have been developed. These include, but no mean complete, slag treatment [3], plasma treatment [4], acid leaching [5], directional solidification [6], solvent refining [7], and so on. Among these, solvent refining is a promising technique for SoG-Si feedstock production. It demonstrates the potential to remove almost all impurities in MG-Si, including B and P elements, relying on the decrease of segregation coefficients of impurities due to the lowered liquidus temperature [8].

Several alloy systems have been developed to purify the MG-Si via solvent refining solution, such as Si-Cu[9, 10], Si-Fe[11–13], Si-Ca[14], Si-Sn[15, 16], Si-Al[17, 18]. Because Al-based alloys have been extensively investigated [19–21] and plenty of valuable results have been obtained in theory and processing techniques, Al-Si alloy has been exploited and validated as one of the appropriate alloy systems and being developed the advanced processing techniques [22, 23]. The refining process could be divided as follows: alloying MG-Si with Al, solidifying the Si phase, and separating the primary Si. There in general are two requirements that should be satisfied for this process. One is the purity of refined Si crystal, which is associated with the photoelectric conversion efficiency in the final cells; the other is the recovery of primary Si dendrites, which is related to the production costs of feedstocks.
Some reported researches have been shown that most of the metal impurities could be removed effectively, owing to the declined segregation coefficients by one or several orders of magnitude in Al-Si alloy [9, 16, 17]. The residual metal impurity concentrations for the purified silicon that was employed die casting scraps as starting materials were below 1 ppmw, except for Al, meeting sufficiently the specification of SoG-Si [24]. Moreover, removal of boron and phosphorous impurities that are generally considered as hardly removal impurities since their high segregation coefficients could be also exerted, either by adding trace amounts of Hf [25] or Ti [26] to Al-Si alloy or by lowering the cooling rate of Al-Si melt [27].

Since the density difference between Si grains and eutectic melt (Al-Si) is not sufficiently large (approximately 0.1 g/cm³), the separation of primary Si dendrites via natural gravity was proved ineffectiveness [28]. Accordingly, several collecting techniques have been carried out to recover primary silicon dendrites, such as supergravity separation [29, 30], heavy medium separation [28], and solidification under an electromagnetic field [22]. Using these techniques, the purified Si crystals could be recovered from Al-Si alloy, probably followed by acid leaching for the last two techniques. However, it is unavoidable that fine silicon particles will be lost during the collecting process. Consequently, larger primary dendrites should be grown during the refining solidification process, which could be approached either by lowering the cooling rate [31, 32] or increasing the Si content in the Al-Si alloy. Based on the mass conversation law, the latter method is more effective. Ullah Mohammad et al. [33] characterized the morphology evolution of primary Si in the Al-Si alloy with Si content in the range of 17–38 wt% after solidification, from fish-bone like for Al-17 wt%Si to plate-like for Al-Si alloy with Si content higher than 38 wt%. Gumaste et al. [34] analyzed the effect of Si content on the recovery rate of primary Si. The results showed that the optimum alloy composition is Al-35 wt% Si for the investigated Si content of 20–50 wt% in the alloy, having a recovery rate of primary Si of 58.8%, which is as high as 99.42% of the theoretical one. Bai et al. [35] found that the content and the size of the primary Si flakes increased with increasing the Si content in Al-Si alloys with Si content from 20 to 40%, indicating more primary Si grains could be obtained in the Al-Si alloy with high Si content. Meanwhile, a monotonically increase of the recovery rate of primary Si as increasing Si content from 25–65 wt% could be obtained in the work carried out by Li and coauthors [29].

To do so, an extremely wide range of Si content was subject to investigation in the Al-Si alloy solvent refining process. A series of samples with various Al-Si alloy compositions (Si content from 30 wt% to 70 wt%) was prepared. The morphology and recovery rate of Si dendrites was analyzed, and then the optimum alloy composition was determined. Based on the obtained results, the growth model of Si dendrites as increasing Si content was proposed.

### 2 Experimental Details

The starting materials were MG-Si lumps and commercial Al powder (2 N, 200–400 mesh), which were blended to form Al-xwt.% Si (where x is 30, 40, 50, 55, 60, 70) mixtures. Approximately 30 g mixtures for each composition were put in a corundum crucible and then heated to 1450 °C and held for 2 h in a SiC electric resistance furnace (GSL-1600X of MTI, Hefei) under a flowing Ar-4 %H₂ atmosphere to form alloy melts. Then, the alloy melts were cooled down to 600 °C with a constant cooling rate of 3 °C/min and held for 2 h. Afterwards, the power of the furnace was turned off, and the samples were treated by furnace cooling.

The obtained ingots were cut along a vertical direction to form two parts using a diamond wire cutting machine (STX-603, MTI). One part was polished and slightly leached with HCl solution (6 mol/L) to analyze the microstructure using the Metallographic Microscope (ZMM-500, Zhoushan, China) and scanning electron microscope (SSX-550, SHIMADZU). The macrostructure of ingots was scanned by Canon scanner (FAX-L1418SG, Canon), and the length and width of primary Si dendrites were statistically analyzed by IPP (Image-Pro plus) software. In this paper, the primary Si grain was defined as larger than 0.5 mm in size. As illustrated in Fig. 1, the defined primary Si was marked by red, while the grains with no more than 0.5 mm in size were not counted (as grains marked by yellow circles in Fig. 1).

The other part of the ingot was acid leached to separate and collect the primary Si crystals. The acid leaching process was as follows: first, the sample was immersed in hydrochloric acid solution (6 mol/L) for 6 h to dissolve the metal Al; then, the Si crystals were leached in aqua regia solution for 6 h to
remove other metals and compound phases, and third was in dilute hydrofluoric acid solution (0.5 mol/L) for 1.5 h to react with SiO₂ phase. These three leaching processes were performed at 298 K. After leaching, the eutectic phase almost dissolved into the solution to ensure no Al-phase residual on the primary Si and eutectic Si (shown in Figs. 2 and 4). The collected Si particles were sieved by a 35 mesh standard sieve (0.5 mm) to separate the primary Si and eutectic Si. Based on the Al-Si phase diagram and the mass of collected primary Si, the theoretical and experimental recovery rates, \( \eta \), could be calculated following Eqs. (1) and (2).

\[ \eta_{\text{theoretical}} = \frac{f_x}{x\%} \times 100\% \]  
\[ \eta_{\text{experimental}} = \frac{m_x}{M_x \times f_x} \times 100\% \]

where \( f_x \) is the fraction of precipitated Si phase calculated by the lever law from the Al-Si phase diagram, \( m_x \) is the weight of collected primary Si after sieving, \( M_x \) is the total weight of Si raw materials, \( x\% \) is the Si content in Al-Si alloy.

### 3 Results

The morphology evolution of primary Si dendrites for Al-Si alloy with Si content from 30 wt% to 70 wt% is depicted in Fig. 2. Since the investigated alloy composition is addressed at hypereutectic alloy, the shape of primary Si appears plate-like structure, which is a characteristic morphology for the precipitated grains in hypereutectic Al-Si alloy with high Si content [33]. This morphology shows some difference with that formed from Si-Fe alloy which demonstrates spheroidizing trend for Si dendrites [12]. Meanwhile, it is clear from Fig. 2(a) that, for the Al-30 wt% Si alloy, the primary Si dendrites distribute homogeneously in the whole eutectic matrix, some of them are more than 10 mm in length. The needle-like Si grains seen from microstructure have the plate-like morphology after acid leaching (seen from the inserted photo in Fig. 2(a)). As increasing the Si content in Al-Si alloy, the primary Si dendrites become larger, appearing thicker plate-like morphology (shown in the inserted photo in Fig. 2(e)). Moreover, more silicon grains exist with increasing the Si content in Al-Si alloy. Therefore, it could be concluded that the primary Si dendrites become larger and denser in the alloy with high Si content after solidification.

To analyze the morphology evolution of primary Si dendrites for Al-Si alloys with various silicon contents, the length and width of silicon dendrites were calculated through the IPP software (mentioned in Section 2). Herein, the primary Si was defined as grains larger than 0.5 mm in size. All grains on the whole vertical section surface for each sample were marked and countered to improve the calculation accuracy, thereafter the average values were obtained. The effect of Si content on the length and width of primary Si dendrites is depicted in Fig. 3. It can be seen that the average width of primary Si dendrites increases gradually from 0.54 mm to 1.23 mm with increasing Si content from 30 wt% to 70 wt% in Al-Si alloys. This result indicates that the thickness of Si dendrites becomes larger in the Al-Si alloy with high Si content, which is beneficial for improving the recovery rate of the refined Si [23]. Moreover, the average length of primary Si dendrites demonstrates a similar tendency in the content range from 30 wt% to 55 wt%. This result is consistent with the conventional understanding that the Si size increases with increasing initial Si content in a hypereutectic Al-Si alloy [36]. However, further increasing the Si content (> 55 wt%), the average length of primary Si dendrites decreases inversely. This is maybe caused by the increased grain density in the Al-Si alloy with

![Fig. 2](image-url) Macrostructures of sectioned Al-Si alloy ingots with various Si contents: (a) Al-30wt% Si, (b) Al-40wt% Si, (c) Al-50wt% Si, (d) Al-55wt% Si, (e) Al-60wt% Si, (f) Al-70wt% Si. The inserted pictures in (a) and (e) are primary Si grains for Al-30wt% Si and Al-60wt% Si alloys after acid leaching, respectively.
higher Si content, which inhibits the grain growth along the length direction. Consequently, higher Si content in Al-Si alloy has a detrimental effect on the growth of primary Si dendrites along the length direction. Undoubtedly, it could be deduced that higher initial Si content (> 55 wt%) in the Al-Si alloy, will influence the growth pattern of the primary Si dendrites. This will be discussed in detail in the next section.

Figure 4 shows the morphology of eutectic microstructure for (a) Al-30wt%Si, (b) Al-55wt%Si, (c) Al-70wt%Si alloys, and the collected eutectic silicon particles (d) and (e) and corresponding morphologies (f) and (g) for Al-30wt%Si and Al-70wt%Si alloys after acid leaching, respectively. It can be seen from Fig. 4(a) that, for Al-30wt% Si alloy, the eutectic silicon has an irregular shape, but aligning almost a line in the matrix. After leaching and sieving, fine eutectic silicon particles could be obtained, as shown in Fig. 4(d). The real morphology of eutectic Si particles is presented in Fig. 4f, showing lamellar shape with a thickness of about 15 μm. This is maybe caused by the fast growth during eutectic alloy solidification. As increasing the Si content in Al-Si alloy, there is no obvious difference in the shape and distribution for eutectic silicon grains. However, some large particles (marked by A and B in Fig. 4b and c, respectively) exist in Al-Si alloys with high Si content (55 wt% and 70 wt%), with the diameter of about 500 μm (shown in Fig. 4g). It could be reasonably deduced that the large particles are the broken part from Si dendrites. Accordingly, the main difference of eutectic Si particles for Al-Si alloys with various Si contents is the fraction of large broken Si particles, i.e., more broken Si particles exist in alloys with higher Si proportions.

The solvent refining process must achieve a recovery rate of refined Si as high as possible, aiming at the reduction of the production cost. The theoretical and experimental recovery rates were calculated according to Eqs. (1) and (2), and the results are displayed in Fig. 5. From the results of the
calculated theoretical recovery rates, it could be found that the theoretical recovery rate increases gradually from 66.4 to 93.8 % with increasing the Si content from 30wt.% to 70wt.%. In the present work, the defined primary Si grain size was larger than 500 μm, so that some fine primary Si particles were inevitably lost during the sieving and collecting process. Accordingly, the experiment recovery rate is lower than the theoretical values. As can be seen from Fig. 5, the experimental recovery rate increases from 44.6 % for Al-30wt.% Si alloy to 83.7 % for Al-55wt.% Si alloy, which could account for 94.9 % of the theoretical recovery rate. The obtained experimental recovery rate is approximately equal to that reported in reference [34]. However, further increasing the Si content of the alloy (> 55wt.%), the experimental recovery rate of primary Si dendrites decreases. When the alloy composition is Al-70wt.% Si, the experimental recovery rate drops to 68.2 %. This tendency is consistent with the length evolution for the Al-Si alloy. Thus, the changed growth pattern influenced the recovery rate of refined Si. Given production cost reduction, the optimum alloy composition is Al-50 – 55 wt% Si.

4 Discussion

Based on the above results, it is clear that the growth pattern of Si primary dendrites has been changed as increasing the Si content in the Al-Si alloy. Generally, on solidification of the Al-Si hypereutectic alloy, the growth of plate-like Si primary dendrites lies on (111) planes and in [211] directions, where it occurs by the twin plane re-entrance edge (TPRE) mechanism [37]. Figure 8(a) illustrates schematically the growth model of the plate-like Si primary dendrites. In the case of Al-30wt.% Si alloy, the Si primary dendrites grew following this model, forming a thin plate-like morphology (shown in Fig. 2(a)). Due to the fragility of pure silicon or by over-energetic sieving, some thin plates were believed to be broken into fine particles (< 0.5 mm) after washing and sieving, which were classified as eutectic Si. This may be the reason why the experimental recovery rate is only 67.2 % of the theoretical value.

Increasing the Si content, more Si atoms are supplied. As solidification proceeds, Si atoms diffuse to the growth front and are trapped by grain surfaces. One possibility is that the Si atoms are located at twin grain edges associated with the TPRE growth mechanism, resulting in a fast growth along < 211 > direction (length direction). The other possibility is that they are located at flat surfaces associated with the lateral growth mechanism [33], resulting in a growth tendency along < 111 > direction (width direction). For the Al-Si alloy with Si content below 50 – 55wt.%, both the length and width of the Si primary dendrites increased with increasing Si content, as shown in Fig. 3. This could be named coarsening process of the primary Si dendrites. The coarsened Si plates were not easily being broken during sieving and washing, thereby improved the experimental recovery rate up to 94.9 % of the theoretical recovery rate for the Al-55wt.% Si alloy.

Further increasing the Si content to more than 55wt.%, the average length of the Si primary dendrites decreased (shown in Fig. 3), indicating the advantage of the preferred growth along < 211 > direction was hindered. From the result of Fig. 2(e)-(f), more Si primary dendrites appeared, i.e. higher grain density in alloys with Si content of 60 – 70wt.%. During solidification, grain growth competed with each other. As the growing front with < 211 > growth direction met neighboring grains, the growth was hindered. This hindering effect would be enhanced with increasing the Si content, resulting in the decrease in length of the dendrites. However, the growth along < 111 > direction was not hindered since the typical two-dimension structure of the dendrites. Thereby, a multi-layered structure formed for the Al-60wt.% Si alloy (illustrated in Fig. 6), leading to a further increase in thickness.

Generally, the plate-like Si primary dendrites have a sharp tip morphology along < 211 > fast growth direction [37], which would be easily broken due to its fragile property, especially surrounded by plenty of neighboring grains during solidification. The broken tip usually has a small size (< 0.5

Fig. 5 The theoretical and experimental recovery rates of primary Si dendrites as a function of Si content

Fig. 6 A multi-layered structure of primary Si in Al-60 wt% Si alloy
mm, as shown in Fig. 4(b) marked as A), which could not be collected after leaching, resulting in a consumption of the refined Si. On the other hand, some crystals were peeled off from the coarsened or multi-layered dendrites due to extremely high thermal stress, as like the broken part marked B in Fig. 4(c), leading to the other type of consumption. These two kinds of broken crystals contributed to the reduction of the experimental recovery rate for alloys with a Si content range of 55 ~ 70wt.%. For further analysis, Si particles with a size range of 0.3 ~ 0.5 mm were employed to characterize the broken crystals, and the statistical results are shown in Fig. 7, where the weight fractions of broken crystals to the total defined eutectic Si (< 0.5 mm) for Al-55wt.% Si, Al-60wt.% Si, and Al-70wt.% Si alloys were calculated, respectively. It can be seen from Fig. 7 that the weight fraction of broken crystals increases with increasing the Si content. The consumption of refined dendrites increases with increasing Si content due to more broken tips from higher grain density and more peeled-off particles from thicker dendrites, named the broken process. Thus, it can be concluded that the broken process has a detrimental effect on the recovery of the refined Si.

Consequently, we could summarize the growth pattern of the primary Si dendrites in the Al-Si hypereutectic alloy with various Si contents, as schematically shown in Fig. 8. In the investigated content range, i.e., 30 ~ 70wt.%, the growth of plate-like Si primary dendrites was dominated by the TPRE mechanism. For the Al-30 ~ 50 wt% Si alloys, the Si primary dendrites grew largely along < 211 > and < 111 > directions with increasing the Si content, i.e., the primary dendrites underwent a coarsening process, increasing the experimental recovery rate of the refined Si. The highest recovery rate could be achieved for Al-50 ~ 55 wt% Si alloys. In the case of Al-55 ~ 70 wt% Si alloys, however, the growth along < 211 > direction was inhibited. Meanwhile, the broken effect originated from grain collision and thermal stress was enhanced as further increasing the Si content, thereby leading to a decrease in the experimental recovery rate.

From the view of the production cost reduction, the optimum composition of the Al-Si alloy for solvent refining has been determined as Al-50 ~ 55 wt% Si, for which a desirable practical recovery rate, as well an attractive impurity removal efficiency of refined Si, could be obtained. The purity analysis will be detailed discussed elsewhere.

5 Conclusions

A series of Al-Si hypereutectic alloys with Si content of 30 ~ 70 wt% were employed to investigate the effect of Si content on the evolution of grain morphology and recovery rate during the Al-Si alloy solvent refining process. The results showed that the plate-like Si primary dendrites grown following the
twin plane re-entrance edge (TPRE) mechanism formed in all alloy compositions. For the Al-30 ~ 50wt.% Si alloys, the morphology evolution of Si primary dendrites was related to a coarsening process by growing along the preferred $<211>$ and $<111>$ directions as increasing the Si content in the alloy, leading to an increase in the experimental recovery rate of the refined Si. When the Si content was at 50 ~ 55wt.%, the coarsened Si primary dendrites resulted in an extremely high recovery rate, up to 94.9 % of the theoretical recovery rate. However, further increasing the Si content to 55 ~ 70 wt%, the preferred growth along $<211>$ direction was inhibited. Some broken crystals caused by grain collision and thermal stress appeared after solidification, named as the broken process of Si primary dendrites, leading to a decrease in the recovery rate of the refined Si. Based on the consideration of production cost reduction for Al-Si alloy solvent refining solution, it could be concluded that the optimum alloy composition is Al-50 ~ 55wt.% Si. In the following work, the effect of Si content on the impurity removal should be illustrated.

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Data Availability Not applicable.

Declarations

Conflicts of Interest/Competing Interests The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Consent to Participate All authors happily agree to participate in this research study.

Consent for Publication All authors permit the permission to the journal to publish this research study.

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