

Influence of direct electric current on solidification process of Al–Si alloy



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ABSTRACT

The effect of direct electric current on redistribution and microstructure of primary silicon in Al–Si melt during the solidification refining process was studied. Electric current generates a Joule heat, which slows down the cooling rate of the furnace. The precipitated area of primary silicon decreases from 100% to 36.67% with the increase of current density from 0 A/m² to 1.5×10⁶ A/m². Furthermore, direct current causes a convection leading to the migration of primary silicon in the opposite direction of electric field, which forms the accumulation area. The morphologies of primary silicon and the redistribution of B impurity were investigated under the effect of electric current.

1. Introduction

With the rapid development of photovoltaic industry, many new potential appropriate methods of refining and recycling solar-grade silicon (SoG-Si) are under research [1]. Recently, alternative methods based on metallurgical purification techniques [2], such as acid leaching [3], directional solidification [4], plasma treatment [5], vaporisation refining, slagging [6] and solvent refining, have been carried out to lower the production cost of SoG-Si. A large amount of works are also focused on solvent refining [7–9], which is a novel refining technique that uses a Si-based solvent at low temperature. The novelty mainly relies on the different segregations of impurities between the melted and solid silicon at low temperature. Al is an effective solvent [10–12]. In solvent refining, MG–Si is alloyed with Al to obtain an Al–Si melt, and Si precipitates from the melt at a temperature lower than the melting point of Si (1687 K); this step can remove most impurities, including P and B, from solid silicon, and effectively reduce the energy cost.

However, several difficulties were encountered in this process. Si crystal is highly dispersed in the solidified Al–Si alloy [13], thereby causing the difficulty to remove inter-grain contaminants and collect the refined Si via acid cleaning. The silicon should be gathered to obtain a high density area of silicon crystal before acid cleaning and consequently improve the recycling efficiency of silicon. Therefore, separation of primary silicon and solvent melt is an inevitable problem in the sustainable development process of alloy refining. Electromagnetic stirring [10,14] and super gravity [15,16] technologies are applied to the solidification process of solvent refining. Flow convection induced

by electromagnetic stirring and super gravity shows a positive effect on the segregation of impurities and collection of silicon. The effect of electric field on alloy solidification has been researched for many years, and most research focuses on solid-state materials and solidification of aluminium alloy under alternate and direct currents [17] and high-current density electro-pulsing field [18]. According to Jiang [19], electric fields show considerable influence on grain size, thereby indicating the acceleration of atom diffusion in liquid, which decreases the lamellar spacing and grain size. However, the research about the effect of electric current on the growth of primary phase and change of temperature field in the solidification process is limited. In the present research, we studied the effect of different direct electric current densities on the solidification of hypereutectic Al–Si alloy. The different resistivity between solid Si and alloy melt result in diverse performance in the solidification process of Al–Si alloy under direct electric current.

2. Experimental methods

The initial alloy composition was Al–28.51 wt%Si (liquid temperature is 1073 K) to achieve low refining temperature. The master alloys were prepared by melting the Al (99.9%; B content, 24 ppmw) and Si (99.9%; B content, 14.8 ppmw) in an alumina crucible under argon atmosphere using an induction furnace. The alloys were quenched into a quartz tube with 5 mm inner diameter by using a vacuum filling technique and cooled in water to avoid macro-segregation.

A horizontal resistance furnace was used for solidification experiments of Al–28.5 wt%Si alloy, as shown schematically in Fig. 1. The master alloys were located in the middle of alumina tube with 5 mm

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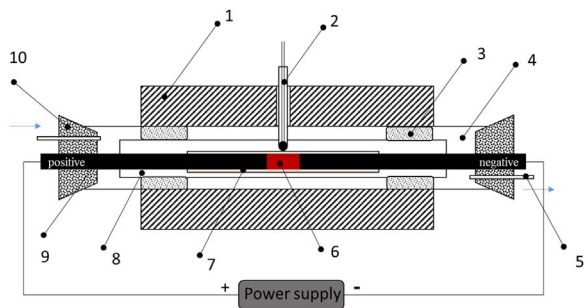


Fig. 1. Schematic diagram of the experimental apparatus, 1-resistance furnace, 2-thermocouple, 3-refractory bricks, 4-quartz tube, 5- gas outlet, 6- Si-Al melt, 7- alumina crucible, 8-quartz tube, 9-graphite electrodes, 10-rubber plug.

inner diameter and 300 mm length. Two graphite rods were placed at two ends of alloy as electrodes. Therefore, both ends of the sample were connected to the graphite electrodes pole for power supply respectively. The system was backfilled in flowing argon at a flow rate of 200 ml/min to protect the graphite electrodes from oxidising in high temperature. Afterwards, the alloys were heated to 1073 K at a rate of 6 K/min and re-melted in a cylindrical resistance furnace. Direct electric current with different densities (5×10^5 , 1.0×10^6 and 1.5×10^6 A/m²) was generated by the power supply (TN-KGZ01). The temperature was maintained at 1073 K for 120 min; subsequently, the furnace was cooled to 723 K, which was below the eutectic temperature (850 K). Finally, the direct electric current was cut off, and the samples were quickly quenched in water to room temperature.

The solidified sample was sectioned longitudinally along the mid-plane. The selected section was ground on SiC paper and polished to a size ranging from 6 μm to 1 μm. The surface morphology of the primary Si plate was examined via metallurgical microscopy (MEF4). Electron probe microanalysis (EPMA-1600, Shimadzu, Japan) was used to detect the redistribution of B in the alloy. The refined Si powder was obtained through acid leaching, whose steps were as follows. Firstly, the alloy was treated with 6 mol/L HCl at room temperature for 64 h to dissolve Al and Zn. Secondly, primary Si platelets were collected, crushed and leached with aqua regia at 333 K for 3 h to remove the residue metals. Afterwards, the Si powders were treated with HF–H₂O solution (volume ratio of 1:17) to remove the SiO₂ films at 333 K for 3 h. Finally, the refined Si powders were rinsed with deionised water until they reached a neutral state and dried. The B concentrations were analysed by using an inductively coupled plasma mass spectrometry (ICP-MS ELEMENT 2, Thermo Electron, Bremen, Germany).

3. Results and discussion

3.1. Effect of direct electric current on the cooling rate of the furnace

According to electromagnetic theory, the thermal effect produced by an electric current going through an alloy per unit time and unit volume can be calculated by using the following equation:

$$Q = i^2 \rho \tag{1}$$

where Q is the Joule heat produced by electric current; i is the density of electric current; and ρ is the electrical resistivity of the alloy. Consequently, the temperature in the alloy and graphic electrode per unit time and unit volume increases, which can be expressed as Eq. (2):

$$\Delta T = \frac{Q}{mC} = i^2 \frac{\rho}{mC} \tag{2}$$

where m is the mass of the alloy per volume, and C represents the specific heat capacity. The increase in temperature of Al–Si alloy and graphic electrode generated by Joule heat at room temperature was calculated and is listed in Table 1. The higher electrical resistivity value of Al–Si alloy (about 10^{-7} Ω m) than that of graphic electrode (about 10^{-6} Ω m) caused the different heating rates per unit volume of the alloy and graphic electrode. The Joule heat energy of the sample decreased the cooling rate in the furnace as shown in Fig. 2. The cooling rate in the furnace was different during alloy refining process; such difference should be attributed to the Joule heat energy of samples obtained with the application of different electric current densities during the cooling process. With the increasing electric current density, the Joule heat energy increased, and the cooling rate in the furnace decreased.

The effects of Joule heat energy on alloy sample and graphic electrodes were also compared. The cooling rate of graphic electrodes was slower than that of the alloy sample during the cooling process. Therefore, the temperature gradient between the two ends and the middle part was established and increased with the increase of electric current density.

3.2. Effect of direct electric current on the Al–Si alloy macrostructure

The samples were connected to the positive electrode on the left end and to the negative electrode on the right end, as shown in Fig. 1. The direct current was imposed on the molten alloy when the temperature reached the liquid temperature (1073 K). The direct electric current

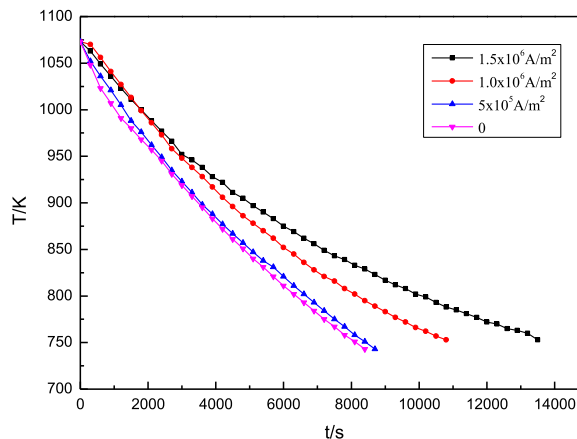


Fig. 2. Cooling rate of the furnace with the application of different current density.

Table 1

The increasing of temperature generated by Joule heat.

Materials	Electric current density (A/m ²)	Electrical resistivity (Ω m)	The mass of the alloy per volume (kg/m ³)	Joule heat energy (J/ s m ³)	Heat capacity (J/ kg K)	ΔT (K/s)
Si-Al alloy	5×10^5	3.95×10^{-7}	2.674×10^3	9.87×10^4	0.904	40.8
	1.0×10^6			3.95×10^5		163.4
	1.5×10^6			8.887×10^5		367.6
Graphic electrode	5×10^5	8×10^{-6}	1.7×10^3	2×10^6	0.71	1.66×10^3
	1.0×10^6			8×10^6		6.63×10^3
	1.5×10^6			1.8×10^7		1.49×10^4

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