



Pretreatment of recycling wiresaw slurries—Iron removal using acid treatment and electrokinetic separation

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ABSTRACT

Iron removal is an important step when recycling silicon wiresaw slurries. The iron to be removed primarily comes from the saw wire generated when cutting silicon ingots. This study assessed the effectiveness of using acid treatment and electrokinetic separation to remove iron from silicon slurries. Experiments focused on the effects of the HNO_3 volume and concentration in the acid treatment and the effects of applied electric field and duration during electrokinetic separation on iron removal efficiency. Experimental results reveal that the operational parameters for optimal removal efficiency were 150 ml of 1 M HNO_3 aqueous solution to remove iron in 40 min via acid treatment, and 1 V/cm and 90 h to achieve a 90.2% removal efficiency using the electrokinetic separation. Via dissolution, cleaning and drying processes in the acid treatment, residual iron ions formed iron precipitates which trapped between Si or SiC particles. However, the metal ions can migrate toward the cathode via the electrical field during electrokinetic separation. Therefore, electrokinetic separation is better at removing iron from wiresaw slurries than the acid treatment.

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1. Introduction

Photovoltaic (PV) technology is a reliable and important source of clean electrical energy [1]. The worldwide PV industry has recently experienced strong economic growth of 40–50% annually. Estimations indicate that the total volume of PV modules produced will be 20 GWP in 2011 [2]. Over 90% of the current photovoltaic industry relies on crystalline silicon cells, which has resulted in a shortage of silicon feedstock. This shortage typically increases feedstock prices significantly. Among the many strategies for improving silicon supply, reuse of rejected silicon, such as recycling silicon from wiresaw slurries, may be an effective solution for overcoming silicon feedstock shortages [3].

In silicon wafer manufacturing, silicon ingots must be cut by multiple wiresaws with an abrasive slurry composed of silicon carbide (SiC) particles in a glycol-based solution. Depending on wafer thickness, kerf loss is 25–50% of the silicon ingot. This silicon kerf drains away with the slurry. The slurry is typically composed of abrasives, solvents and steel residue from the wires [4]. The silicon content in sawing waste can be as high as 40% by weight. Therefore, a sufficient amount of silicon can be obtained for current

silicon solar-cell production if this silicon can be purified from sawing waste [3,5].

Silicon sawing waste contains many tiny iron fragments generated from broken sawing wires during cutting. These fragments make the Si separation process difficult and degrade the performance of reused Si or SiC. For examples, recovered SiC abrasives attached to iron segments weaken cutting performance [6], and iron impurities in the recovered silicon reduce the lifetime of solar cells [7,8]. Therefore, when recycling SiC or Si from wiresaw slurries, iron impurities must be removed. Wang et al. [9] successfully developed a novel process for purifying the silicon obtained from slurry; this process comprises chemical pretreatment, centrifugation, high-temperature treatment and directional solidification. The objective of the pretreatment step is to remove iron impurities from wiresaw slurries. During their pretreatment step, an acid solution was used; the removal fraction reached about 0.876. Although residual metal impurities can be further reduced by the subsequent high-temperature treatment, Wang et al. [9] did not investigate the enhancement of iron removal or the residual form of iron in slurries prior to the energy-intensive steps. Herein, we attempted to study different pretreatment methods for recovering SiC or Si from wiresaw slurries.

Acid treatment is a typical method used to remove metal impurities from solids. During acid treatment, the metal dissolves in a solution and thus can be separated from a solid via filtration or centrifugation. Electrokinetic separation is an alternative method for removing metal impurities. This method does not use additional

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chemicals and only requires a power supply and two adequate electrodes to drive the treatment. Under the action of an electric field, the metal can dissolve in slurries via redox reactions. Cations can then be removed by electrokinetic transport [10].

Although acid treatment and electrokinetic separation can be utilized for removing metals, few studies applied these methods for iron removal from wiresaw slurries. This study determined the effectiveness of acid treatment and electrokinetic separation for removing iron fragments from wiresaw slurries. Nitric acid and an electric field were applied to treat wiresaw slurries. The transport phenomena and accompanying reactions in this treatment were examined. Furthermore, to improve metal removal efficiency, the optimum operational parameters were identified.

2. Experimental

To fix the composition of silicon slurries, a mixture of 40 wt.% silicon, 53 wt.% silicon carbide and 7 wt.% iron powder were mixed to form an aqueous slurry. After mixing the slurry well via mechanical agitation, the specimen was transferred to the metal removal processes, *i.e.*, acid treatment and electrokinetic separation.

During acid treatments, 15 g of slurry was added to HNO₃ solutions with different volumes and concentrations for various durations to identify the optimal acid treatment parameters. The volume of the HNO₃ solution was changed from 50 to 300 ml, the concentration was adjusted from 0 to 3 M, and mixing duration was 20–180 min. After the mixture was oscillated in a shaker for a specific duration, the mixture was centrifuged at 6000 rpm for 30 min. Then the upper-layer liquid in the centrifuged tube was analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES, PerkinElmer 3100XL). The measured amount of iron represented the amount of iron removed by acid treatment, and the amount of iron removed relative to the initial amount of iron was used to determine the Fe removal fraction.

Fig. 1 shows the electrokinetic removal cell used in this study. The electrokinetic treatment of silicon wiresaw slurries was conducted in a cylindrical tube 10 cm in length (L) and 2 cm in diameter. After the specimen was filled in the tube, a constant voltage was applied by a power supply (Model 236; Keithley Co.) across the inert graphite electrodes. To investigate the iron removal performance, the electric fields were changed from 0.5 to 5.0 V/cm, and treatment durations varied from 30 to 120 h. Furthermore, a fluid reservoir with a constant hydraulic head (50 cm-H₂O) was connected to the anodic compartment to supply a hydraulic flow. On the other side of the cell, a bottle for collection of effluent was connected to the cathodic compartment. The pH values of the anodic and cathodic compartments were measured *in situ* by pH electrodes. At the end

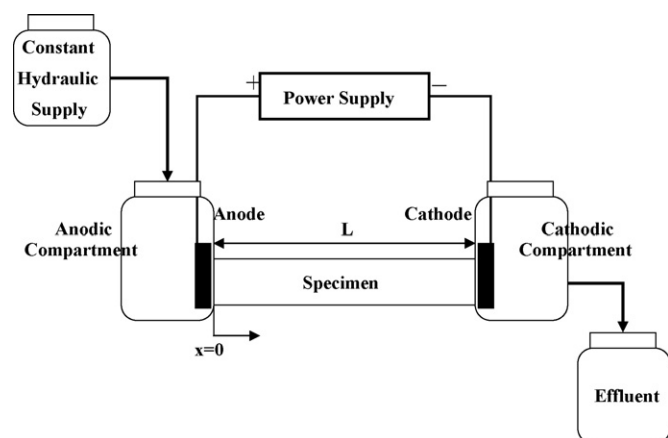


Fig. 1. Schematic diagram of the experimental cell.

of the electrokinetic experiments, the specimen was pushed out of the cylindrical cell and sliced into five equal segments between the anode and cathode, followed by measuring their pH. Each segment was then mixed with 6.0 M HNO₃, and the mixture was oscillated in a mechanical shaker for 24 h and then centrifuged at 6000 rpm for 30 min. Finally, the upper-layer liquid in the centrifuged tube was analyzed by ICP-OES. The residual amount of iron in each segment was then obtained. The ratio of the residual amount relative to the initial amount of iron was used to determine the residual fraction and iron removal fraction.

After iron was removed from slurries by acid treatment or electrokinetic separation, the residual mixture was placed in a vacuum oven at 150 °C to remove moisture. The obtained dry powders were analyzed by SEM, and the residues in specimens were qualified by energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

3.1. Acid treatment

Fig. 2 shows the Fe removal fraction of the 15-g specimen treated by 1.0 M HNO₃ at different volumes. Experimental data show that the Fe removal fraction increased as the HNO₃ volume increased, meaning that HNO₃ removed iron from the specimen and dissolved iron in a solution. The Fe removal fraction increased slightly after adding more than 150 ml HNO₃. This experimental result indicates that the Fe removal fraction reached 0.903 via treatment with 150 ml of 1.0 M HNO₃ for 1 h.

Fig. 3 shows the Fe removal fraction of the 15-g specimen after treatment with 40 ml HNO₃ at different concentrations. Experimental results indicate that iron was not removed by water without HNO₃. However, when the slurry was treated with 0.5 M HNO₃, the Fe removal fraction increased markedly, meaning that the HNO₃ dissolved iron and removed iron from the specimen. When the amount of HNO₃ exceeded 0.5 M, the Fe removal fraction increased gradually as the HNO₃ concentration increased, indicating reduced removal efficiency. Experimental data also indicate that the Fe removal fraction was only 0.711 via treatment with 40 ml of 0.5 M HNO₃ for 40 min.

Fig. 4 presents the relationship between Fe removal fraction and mixing duration. This relationship indicates that the Fe removal fraction increased as mixing duration increased; however, an adequate mixing duration was 40 min. When mixing duration

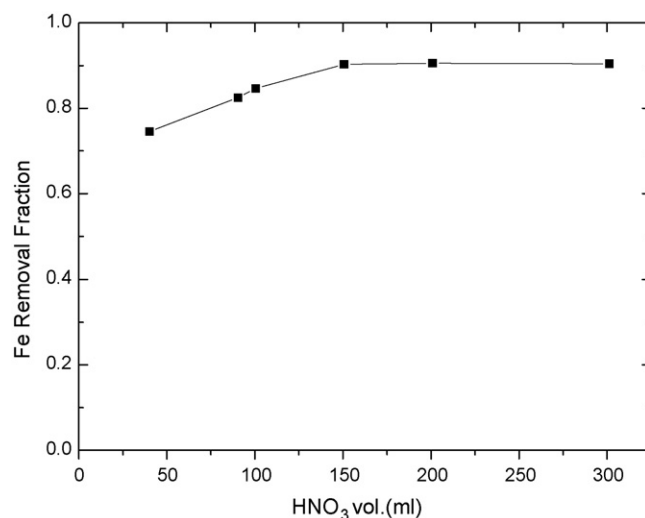


Fig. 2. Fe removal fraction after treatment with 1.0 M HNO₃ at different volumes for 1 h.

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