Journal of Crystal Growth 453 (2016) 143–150

Contents lists available at ScienceDirect

Journal of Crystal Growth

journal homepage: www.elsevier.com/locate/jcrysgro

Recovery of cutting fluids used in polycrystalline silicon ingot slicing

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ARTICLE INFO

Article history:
Received 21 July 2016
Received in revised form
16 August 2016
Accepted 17 August 2016
Communicated by P. Rudolph
Available online 17 August 2016

Keywords:
Slurry
Silicon carbide
Polyethylene glycol
Silicon ingot
Silicon recovery

ABSTRACT

A recovery process for effective separation of silicon, silicon carbide micro powders and polyethylene glycol from the wire sawing slurry is proposed. The separation between silicon and silicon carbide is based on their size difference and surface charging state. The aim of this work is the study of the solid phase and liquid phase separation of silicon carbide and Silicon. Some methods applied for this purpose are the centrifugation process, phase-transfer separation as well as liquid–liquid extraction followed by the regeneration of polyethylene glycol by the distillation process. It is verified experimentally that silicon and silicon carbide micro powder can be effectively separated by phase transfer separation, centrifugation, chemical cleaning, filtering and distillation. In this study, the liquid–liquid extraction was used to separate a particle from a powder mixture. The removal of liquid component from a solution via a solvent separated considerably larger silicon carbide particles. The optimal results showed that Si content can reach 82% in the Si-rich powder and 3.8% wt% in the silicon carbide-rich powder. By separating the mixed powder the content reached 31.2 wt% of silicon, 63.3 wt% of silicon carbide as the raw material, 5.3 wt% of the iron fragment, and 0.2 wt% of other impurities.

1. Introduction

Photovoltaic (PV) is the name of a method of converting solar energy into direct current electricity using semiconducting materials that exhibit the photovoltaic effect. The total proportion of electricity generated by solar cells is steadily increasing and it has attracted many scientists to develop new technologies, among which the polysilicon cell is regarded as one of the fascinating ways to transfer the solar light into the storage electrical energy [1,2]. Silicon is the most widely used material in photovoltaic industries [3,4]. As per current status, the consumption of silicon wafer currently accounts for more than 70% of the cost for solar cells [5,6].

Although, the solar cells causes no pollution [7,8] however, the different technological steps of manufacturing, such as crystal growth, the surface treatment, the cleaning of silicon wafers, the encapsulation devices and in particularly, cutting the ingot into silicon wafer by multi-wire slicing process [9], producing a great amount of hazardous kerf loss silicon with tiny silicon particles, resulting in serious environmental problems [10].

The hazardous kerf loss silicon is in the form of slurry it's a viscous mixture that consists of pure fine particles of silicon (Si), silica (SiO_2), abrasive silicon carbide (SiC) particles, metal impurities from cutting wire, polyethylene glycol (PEG) solution and additives for better particle suspension. Since slurry is one of the more expensive products used in the solar wafer manufacturing process, its recovery and utilization as cutting fluid greatly reduce the amount of waste, thereby the total slicing cost can be efficiently reduced and is a big challenge due to the lack of effective separation and recovery technologies up to now. Our company (CRTSE) invests a large amount of money into this field.

Also, the polysilicon recycled from slurry wastes and resupplied as the silicon feedstock to the field of solar cell fabrication again, it will save the shortage of solar grade silicon materials and bring in the enormous environmental and economic benefit.

Furthermore, SiC, Si particles and metal fragments suspended in PEG liquid are so stable that recyclers have difficulty obtaining a low-turbidity PEG liquid from silicon slurry waste. Therefore, various techniques have been proposed obviously, a satisfactory recovery process should be simple in operation and economically acceptable, such as centrifugal separation and filtration [11], coagulation [12], ultrafiltration [13], ion exchange treatment [14], vacuum evaporation for removing water, and decolorizing for adsorbing impurities [15] and phase transfer separation [16]. However, the suggested methods were only patented, i.e. no practical data could be available for their use in recovering PEG liquid from silicon slurry waste. Consequently, the use of sedimentation process to recover PEG from slurry waste [17,18] is the simplest way to separate solids from liquids and is easily performed in factories, such as the cases for recycling lubricant [17] and heavy oil [18]. Lin et al. [16], developed the phase-transfer separation process to recover silicon powder and they used a

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http://dx.doi.org/10.1016/j.jcrysgro.2016.08.035

heavy liquid with a density between that of Si and SiC as the centrifugation medium. Based on the different densities, surface charge and particle size of the wire sawing Si and SiC powders, Shibata et al. [19], Huang et al. [20], applied froth flotation technology to separate Si and SiC and obtained the quite good results, but the separation details, as well as the extraction principle, were not mentioned at all. Wang et al. [21] reported the recovery of silicon from kerf loss slurry wastes using a process including chemical treatment, heavy fluid-gravity centrifugation, high-temperature treatment, and directional solidification. Later, Wu and Chen [22] and Tsai [23] investigated the effect of electrical fields, operation time, and baffle plates on the separation of Si and SiC.

In the present study, phase transfer separation as liquid–liquid extraction process coupled with centrifugation and distillation have been proposed to effectively recover cutting fluid, which is based on the different particle size and surface charge in the aqueous solution. This process is the simplest way to separate solids from liquids and is easily performed in factories, such as the cases for recycling lubricant and heavy oil [24]. To obtain a low-turbidity PEG liquid from silicon slurry waste, this study employed different solvents (trichloroethane, chloroform, etc.) as solvent extracting. Observation of the settling behaviors revealed the separation mechanism of particles during recovery of PEG liquids from silicon slurry waste. Finally, the recovering process to obtain a clean PEG liquid was built up.

2. Theoretical aspect

2.1. Silicon solar cell fabrication process

The raw material of most solar cells today is crystalline silicon. Luckily, silicon is one of the most widely available elements in the form of sand. Before silicon can be cut into thin wafers, however, it has to be purified, as otherwise, the photo effect will not be very efficient. The production of a typical silicon solar cell (Fig. 1) starts with the carbothermic reduction of silicates in an electric arc furnace. In this process, large amounts of electrical energy break the silicon–oxygen bond in SiO_2 via an endothermic reaction with carbon. Molten Si–metal with entrained impurities is withdrawn from the bottom of the furnace while CO_2 and fine SiO_2 particles escape with the flu-gas. Metallurgical grade silicon (MG-Si) at about 98.5% purity is sold to many different markets. Purity levels for solar cells do not have to be as high as in chip applications. Solar-grade purity is 99.999% (5 N) as opposed to electronic-grade silicon purity of up to 99.9999999% (9 N) [25,26].

The majority of MG-Si is used for silicon and aluminum alloys. A much smaller portion is used for fumed silica, medical and cosmetic products and micro-electronics. A small but rapidly growing portion is used for solar applications [25,26].

2.2. Principles of wafer slicing using slurry

The current process for making solar modules in our Research Center in Semiconductor Technology for Energetics (CRTSE) involves four key steps: polysilicon production, ingot shaping and wafering, cell production and module production. This article will focus specifically on the wafering step. The wafering process involves slicing the polysilicon ingot into thin wafers with diamond wire (i.e;. A multi-wire saw with polyethylene glycol, containing silicon carbide (SiC) abrasives, is used as vehicle during the processing) [27]. The slicing is done by running a thin steel wire coated with a SiC and PEG through the ingot (Fig. 2). Wire saws shape the ingots into square blocks which dimensions are about 150 cm × 150 cm × 60 cm. The large casting normally weighs 240 kg before it is sawed into bricks. Later, the bricks are sliced into thin wafers which are used as the base for the active PV cell. The wire is arranged in such away that the ingot is sliced into hundreds of wafers simultaneously. During the slicing operation, moving wire carrying abrasive slurry to create the cutting action is used so a thin layer of cutting slurry is deposited onto the wire [28–31]. The densities of the carrier fluid can vary from approximately 0.85 kg/L for oil-based fluids to over 1.12 kg/L for PEG. In order to create an effective cutting fluid [29], various ratios of the abrasive powder a reused for the suspending liquid. One common mixture 48 wt% of SiC (F500) combined with 52 wt% PEG. The resulting slurry has a combined density of 1.634 kg/L. The wire serves only as the transport mechanism. The combination of wire diameter, nominal abrasive size, carrier vehicle viscosity, and abrasive shape determine the kerf loss of the cutting process [32,33].

3. Experimental details

A slurry waste is mixture of PEG, scrap metal in fine and large particles (mainly iron fragments), polycritalline-Si and SiC (Fig. 3). The separation of Si and SiC from slurry waste is a hard task because of they own similar properties. SiC has a crystalline appearance with a color that varies from green to black (Table 1), is a chemically very stable compound. SiC it is insoluble in water, acidic and it does not react with nitric, sulfuric and hydrochloric acid, then it's soluble in alkalis, the Fig. 4 show the Polycrystalline-Si and SiC and the chemical characteristic of SiC is shown in Table 1.

The diagram in Fig. 5, summarizes the various treatment steps

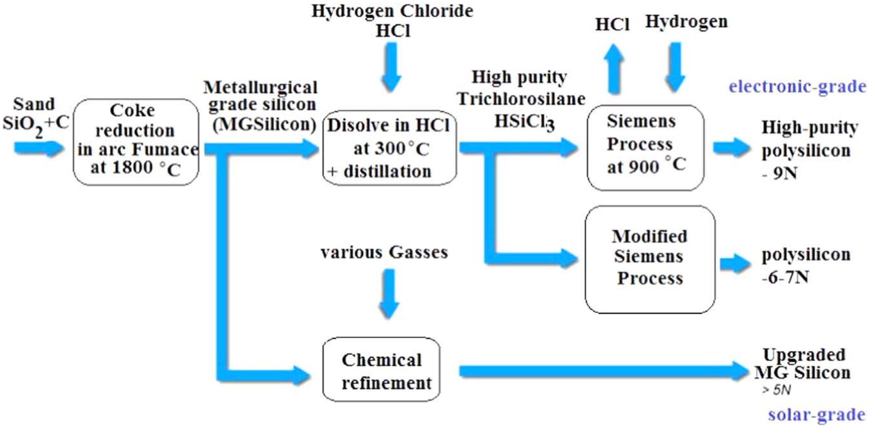

Fig. 1. Manufacturing Silicon steps.