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A rapid thermal process for silicon recycle and refining from cutting kerf-loss slurry waste

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ABSTRACT

More than 40% of the high-purity silicon has been wasted as kerf loss during wafer slicing. This waste not only causes the environmental burden, but also increases the cost for silicon wafers. Although Si could be enriched easily to 85 wt% from the waste, it was extremely difficult to further increase the purity of over 99 wt% due to tiny SiC particles and metallic debris. We proposed a novel rapid thermal process, which was about one hundred times faster than the previous high-temperature treatment, to agglomerate Si in a couple minutes from the pretreated solid powder. With proper conditions, SiC particles and metals could be easily segregated to the surface of Si agglomerates. The high purity Si could be obtained by surface etching, and the best recycle yield was over 70%. The factors, such as temperature, holding time, and the surface oxidation of Si, were further discussed.

1. Introduction

With the awareness of global warming and the depletion of fossil energy, the development of renewal energy has been paid much attention in recent years, especially the silicon-based solar photovoltaics (PV). The rapid development of silicon PV industry, particularly in Asia, has driven down the module cost dramatically, and the grid parity has been reached in a few countries. Unfortunately, the energy payback time for silicon PV is still more than 1 or 2 years, and the key reason is due to the expensive silicon feedstock, which is still produced by the energy-intensive Siemen's process [1]. Ironically, more than 40% of the valuable silicon is thrown away as kerf-loss slurry waste during wafer slicing. The waste also contains the cutting oil, metal impurities and abrasive SiC particles. These materials not only cause the environmental burden but also increase the cost of silicon wafers. Although the cutting oil and SiC particles have been quite successful recycled in industry, the recycle of silicon from the kerf-loss waste remains a big challenge [2–4]. In 2013, more than 30 GW silicon solar cells were produced, and the kerf-loss silicon was estimated to be around 70,000 tons by taking the silicon consumption of 6 g/Wp for solar cells. This also accounted for the waste of 7 TW h electricity based on the silicon feedstock's energy consumption (100 kW h/kg). Nowadays, the total disposal of the slurry waste including abrasive particles and oil are more than 175,000 tons, and they are potentially harmful to the environment. Therefore, to save the cost and to reduce the energy payback time, as well as to protect the environment, the recycle of silicon from the kerf-loss waste is necessary.

The cutting slurry waste after slicing is consisted of the kerf-loss silicon (~30 wt%), SiC (~35 wt%), metallic debris from the cutting wire (<5 wt%), the cutting oil, and the additives for particles suspension. The removal of metals and the majority of SiC particles could be easily achieved by acid leaching and sedimentation [5,6]. Electrical and magnetic forces could also be considered [7–10]. The silicon purity of around 85 wt% could be easily obtained with low cost. However, the further reduction of SiC particles to 1 wt% or less is rather costly [5,11]. Recently, Wang et al. [5,11] proposed a novel high-temperature process to agglomerate silicon particles from the Si/SiC powder mixtures. However, his treatment that needed a few hours was rather time consuming and also energy-intensive. More importantly, the yield was not easy to control because of the oxidation of fine Si powders during heating and melting stages. The best yield was less than 50%.

In this paper, we proposed a much simpler and energy-saving process by using a rapid thermal process (RTP) for silicon recycle. Our process just needed several minutes, instead of a few hours required in the previous high-temperature treatment [5,11]. The process could be easily scaled up due to the short processing time in a normal argon atmosphere. It was found that in some conditions the agglomeration of Si could easily segregate SiC particles and metals to Si surface. Moreover, the recycle yield could be easily controlled, to agglomerate Si in a couple minutes from the

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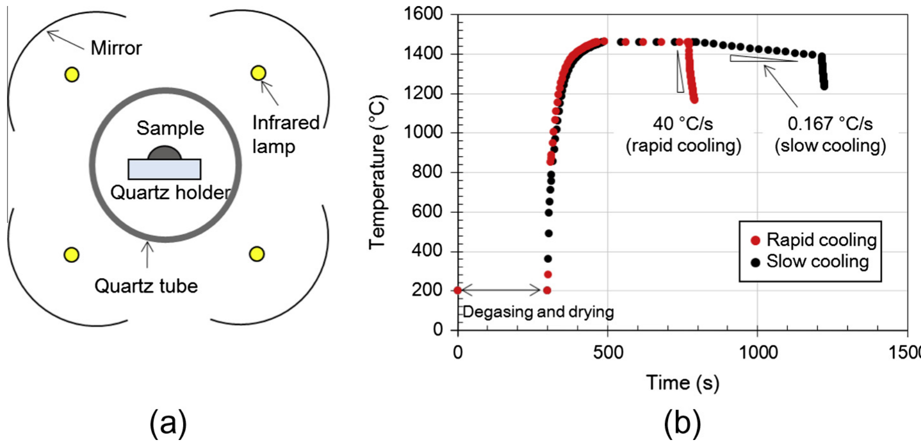

Fig. 1. (a) Schematic of the RTP setup; (b) sample thermal cycles for fast cooling (0.167 °C/s) and slow cooling (40 °C/s); the first 5 min was used for degassing and water removal at 200 °C.

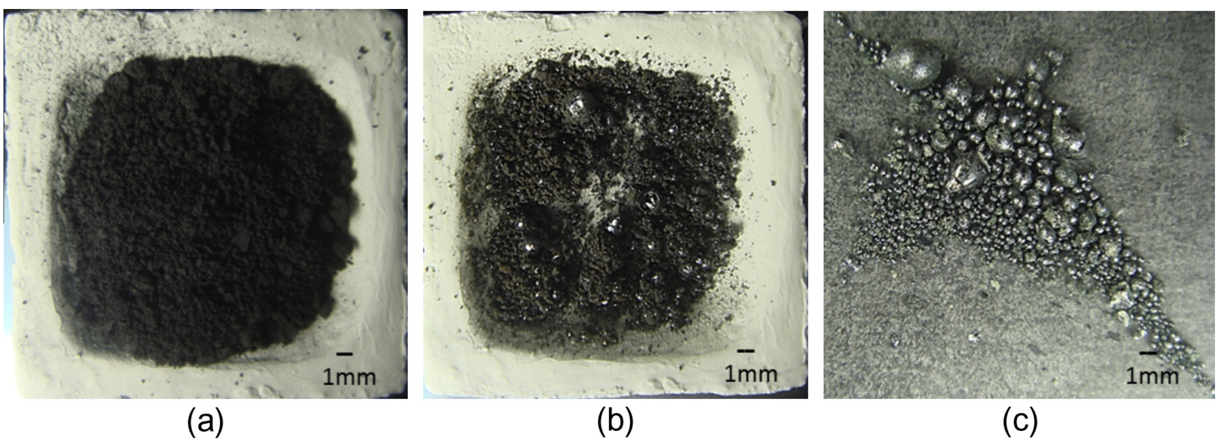

Fig. 2. Images of 85% original Si powder: (a) before RTP; (b) after RTP with slow cooling; (c) the cleaned Si agglomerates.

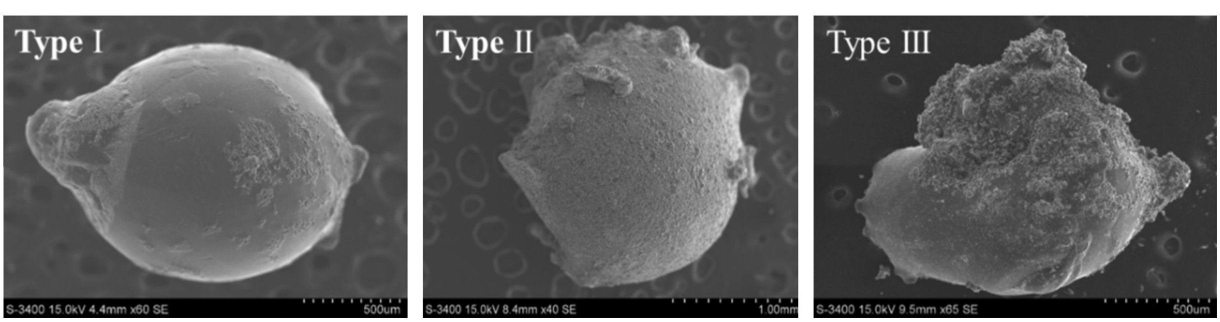

Fig. 3. Three types of Si agglomerates after RTP process.

pretreated solid powder. In the next section, the experimental details are described. Section 3 is devoted to results and discussion, followed by conclusions and comments in Section 4.

2. Experimental

The kerf-loss Si powder used in this study, which was comprised of 85.3 wt% Si, 14.6 wt% SiC particles and 0.1 wt% metallic fragments, was provided by Hong-Tung Inc., a cutting oil recycle company in Taiwan. The original waste was from the used kerf-loss slurry, which contained about 30 wt% Si, during the wafer slicing of solar silicon ingots having a resistivity in the range of 0.5–5 Ω cm. In the company, the waste was first treated by using a filter plate press process to remove oil and a water cyclone to separate large SiC particles to produce "the kerf-loss Si powder". The kerf-loss Si powder was washed with acid and DI water to remove metals and then dried in an oven, hereafter called "original Si powder" in this study.

We proposed a two-stage process to recycle and refine Si. In the first stage, Si was agglomerated from the original Si powder by using RTP. In the second stage, Si was cleaned by acid solution and DI water, and then collected by sieving; in the meantime, SiC