

The removal of saw marks on diamond wire-sawn single crystalline silicon wafers

Kyoung Hee Lee[†]

Department of Biochemical Engineering, Dongyang Mirae University, Seoul 08221, Korea

(Received August 29, 2016)

(Revised September 12, 2016)

(Accepted September 23, 2016)

Abstract The diamond wire sawing method to produce silicon wafers for the photovoltaic application is still a new and highly investigated wafering technology. This technology, featured as the higher productivity, lower wear of the wire, and easier recycling of the coolant, is expected to become the mainstream technique for slicing the silicon crystals. However, the saw marks on the wafer surface have to be investigated and improved. This paper discusses the removal of saw marks on diamond wire-sawn single crystalline silicon wafer. With a pretreatment step using tetramethyl ammonium hydroxide ((CH₃)₄NOH, TMAH) and conventional texturing process with KOH solution (1 % KOH, 8 % IPA, and DI water), the saw marks on the surface of the diamond wire-sawn silicon wafers can be effectively removed and they are invisible to naked eyes completely.

Key words Saw marks, Silicon, Czochralski, Diamond wire saw, Texturing, Chemical etching

1. Introduction

Wire sawing technology has been widely used in manufacturing of brittle-and-hard materials such as silicon, silicon carbide (SiC) and sapphire. The boost of semiconductor and photovoltaic industry has been the primary drive of the development of wire sawing technology as crystalline silicon materials are used as the substrate in those industrial sectors and wire sawing technology provides an effective and efficient approach for wafering. Multi-wire slurry sawing (MWSS) was adopted as the process for semiconductor silicon wafer production as wafer size increased in the 1990s. Compared with other methods (such as Inside Diameter (ID) sawing and Outer Diameter (OD) sawing), the MWSS technique has the advantages of higher throughput, smaller kerf loss and the ability to cut ingots of large size [1].

A schematic of the MWSS system for silicon wafering is shown in Fig. 1. A single stainless steel wire is fed from a supply spool through a pulley and tension control unit to the wire guides.

The multi-wire fixed abrasive diamond wire sawing (DWS) technology has rapidly gained industrial attentions due to its potential for two to three times higher productivity and the potential for kerf recycling. Diamond wires are made by adhering diamond particles to

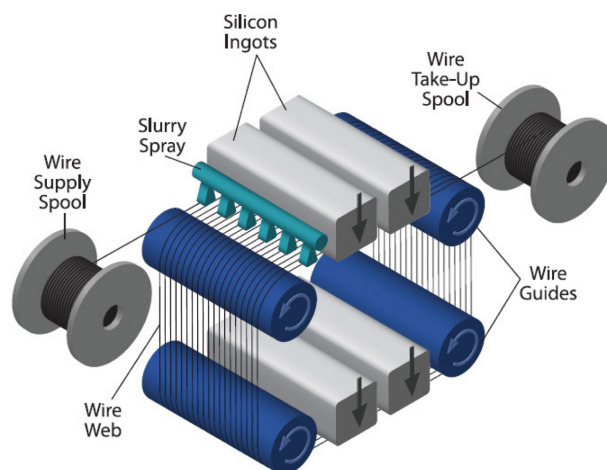


Fig. 1. A schematic of multi-wire slurry sawing of silicon wafers [2].

steel wires using electroplating or resin adhesive. Application of the DWS technology has started to prevailed over MWSS technology in the production of single crystalline silicon wafers for the photovoltaic application in the recent years [3, 4].

Solar power is an attractive alternative source of electricity today. Silicon solar cells convert photons from the sun to electricity. Among the currently available solar cell technologies, silicon crystalline solar cells dominate the market share. A main category of defects found in silicon solar wafers is saw marks. They occur when a silicon ingot is sliced into wafers. Saw marks are a form

[†]Corresponding author
E-mail: lkh@dongyang.ac.kr

of serious saw damage and it becomes a noticeable defect in the wafer surface as the height/depth of a saw mark exceeds 10 to 15 μm in the slicing process [5].

The saw marks generated by DWS cannot be removed perfectly by commercial texturing process for crystalline silicon wafers and they are still left visible from time to time. Although there have been solid evidences indicating that actual roughness of the diamond wire-sawn wafers containing saw marks is less than that of MWSS wafers, and these saw marks should not affect performance of solar cells. Unfortunately, the market will not accept it and the nature of the saw marks on DWS wafers has been studied in recent research [6]. The saw mark problem has not been solved yet. Sand blasting was proposed to remove the saw marks of DWS silicon wafers [7]. In this work, the removal of saw marks on DWS silicon wafers will be studied.

2. Experimental Procedure

Czochralski silicon crystals with a diameter of 200 mm and a length of 2,000 mm were grown using 24 inch hot zone configuration. Conventional boron doping method was used to grow (100)-oriented p-type silicon single crystals. B-doped ingots were sliced to wafers with 170 μm thick and $156 \times 156 \text{ mm}^2$ using DWS technology. Instead of using SiC grits in slurry as cutting agents, the stainless steel wire used in DWS is impregnated or electroplated with diamond grits serving as fixed cutting points. Water based coolant is typically used. Two types of the commercially available diamond wires are shown together in Fig. 2.

Photovoltaic production widely uses potassium hydroxide (KOH) as aqueous texturing solutions. These alkaline solutions etch the silicon anisotropically and form small pyramids on the silicon surface. Hence, light collection is increased by multiple reflections. KOH etch-

ing solutions are cost and time efficient. In this work, texturing solutions were prepared using 1 % KOH, 8 % standard isopropyl alcohol (IPA), and deionized (DI) water. The bath temperature was controlled at 70°C.

Commercial 25 % tetramethyl ammonium hydroxide ($(\text{CH}_3)_4\text{NOH}$, TMAH) was also tested as a pretreatment in order to remove the saw mark perfectly before conventional alkaline texturing process. The bath temperature was controlled at 70°C. All etching experiments were carried out using diamond wire-sawn p-doped wafers. Before any etching process, samples were dipped in a 5 % HF solution for 10 s in order to remove the native oxide, and subsequently rinsed in DI water.

Light reflectivity of the silicon wafers was examined using UV/VIS spectrophotometer and the average reflectivity in the wavelength range of 400–1000 nm was used to represent the reflectivity of the sample.

3. Results and Discussion

3.1. Two different scale saw marks

The image of a typical diamond wire-sawn silicon wafer is shown in Fig. 3. As shown in Fig. 3, the diamond wire-sawn silicon wafers display a shiny surface. The initial reflectance of the diamond wire-sawn wafers is typically higher than that of the slurry processing wafers [4]. The surface of diamond wire-sawn wafer bears remarkable parallel saw marks. They repeat with a cycle of $\sim 0.5 \text{ mm}$. During diamond wire sawing process, the saw wire runs backward and forward repeatedly. It was found that this cycle matched with the width of the cut by a full run of the diamond wire. It is generally believed that these saw marks are caused by running of diamond wire motion back and forth and the parallel pattern of the scratches and the shiny surface of diamond wire-sawn wafers enhance the appearance of the saw marks.

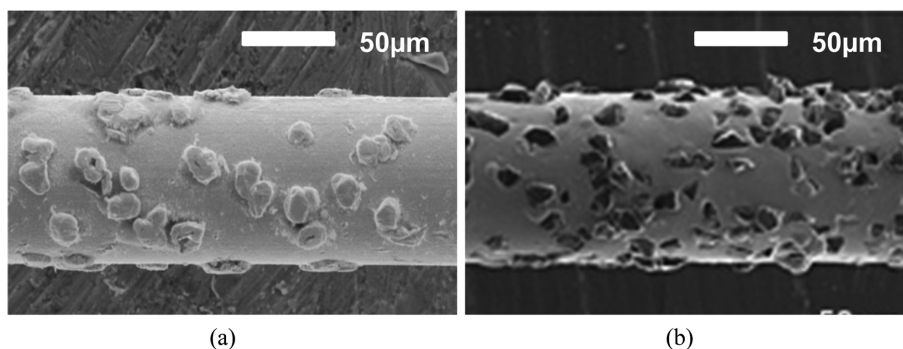


Fig. 2. Scanning electron microscope (SEM) image of diamond wires. (a) electroplated (b) resin adhesive.

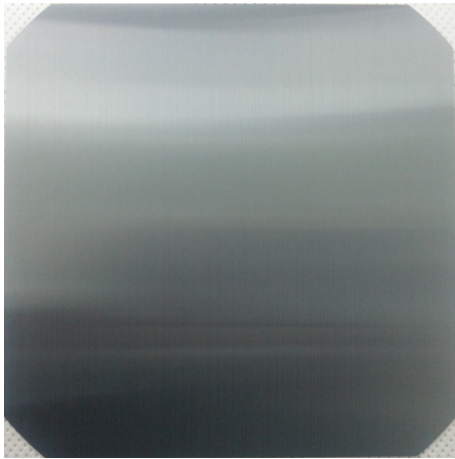


Fig. 3. An image of diamond wire-sawn single crystalline silicon wafer.

This type of millimeter scale saw marks is called periodic fringes.

As shown in Fig. 4, scanning electron micrograph (SEM) of the surface of diamond wire-sawn single crystalline silicon wafer exhibit the micrometer scale saw marks. They are generated by scribing of diamond particles across silicon surface. This type of saw marks is called scratches.

Yu et al. [8] used to find a kind of periodic fringes, consisting of $5.4\ \mu\text{m}$ high and $0.18\ \text{mm}$ spanned ridges, on slurry-sawn silicon wafers using interferometric imaging. But they are invisible to naked eyes completely. The reason for the rougher appearance of the diamond wire-sawn wafers is the visual enhancement of the contrast of roughness by the background of parallel smooth grooves. For the same reason, the millimeter scale fringes

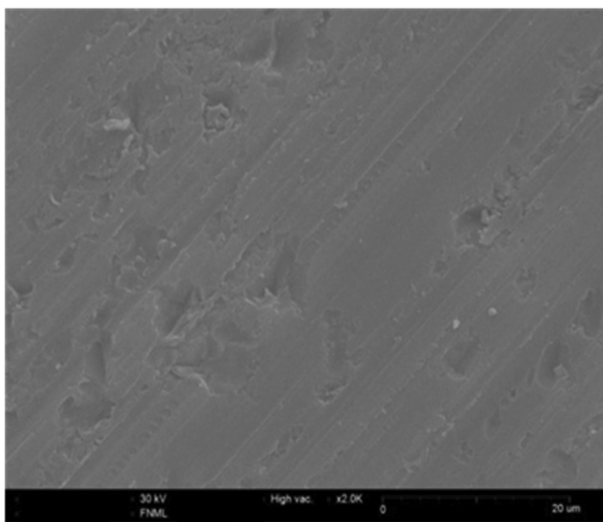


Fig. 4. Scanning electron micrograph (SEM) of the surface of diamond wire-sawn single crystalline silicon wafer.

of slight ridges on the diamond wire-sawn wafers become visible to naked eyes.

3.2. Chemical etching

Alkaline texturing using KOH solution (1 % KOH, 8 % IPA, and DI water) has a strong crystallographic preference over mechanical damage-induced preference, which makes it a prospect in removing the scratches on diamond wire-sawn wafers. Unfortunately, the periodic fringes are still visible. It is difficult to flatten the slight ridges of the periodic fringes at millimeter scale by alkaline etching. In this work, the elimination of the mechanical damage on the surface of diamond wire-sawn silicon wafers using chemical etching with TMAH and conventional alkaline texturing are proposed in order to make the saw marks invisible to naked eyes. We also propose that the saw marks are the visual contrast effect among deep and shallow grooves. The main idea is reducing the visibility of the millimeter scale saw marks by controlling reflectance of diamond wire-sawn silicon wafers.

In order to remove saw marks, a number of pretreatment solutions have been investigated in this preliminary work, such as SC1 (NH_4OH , H_2O_2 , and H_2O), SC2 (NH_3 , H_2O_2 , and H_2O) clean process, and citric acid. Unfortunately, these solutions cannot remove the saw marks to acceptable level. We studied TMAH pretreatment to remove the saw marks using conventional texturing process. Fig. 5 shows SEM images of the surface of diamond wire-sawn single crystalline silicon wafers after pretreatment in TMAH for different times from 10 min to 50 min. With the increase of pretreatment time, the saw marks are erased, and totally removed after 50 min in micrometer scale. Some of facets appear and get large during the pretreatment step. The pretreatment step is useful for erasing the micrometer scale saw marks. The surface layer of silicon wafers have removed, but the periodic fringes are still visible to naked eyes sometimes.

The diamond wire-sawn single crystalline silicon wafers after 50 min pretreatment using TMAH are applied conventional texturing process using KOH solution. The saw marks on the surface of textured diamond wire-sawn single crystalline silicon wafers become invisible to naked eyes or completely removed. The measured light reflectivity of the surface of TMAH pretreated wafer is shown in Table 1. A typical value of as-cut wafer is presented in the table for the reference. As can be seen in the table, a remarkable improvement in the reflectivity reduction is obtained with TMAH pretreatment. The light reflectivity is reduced to as low as 10%

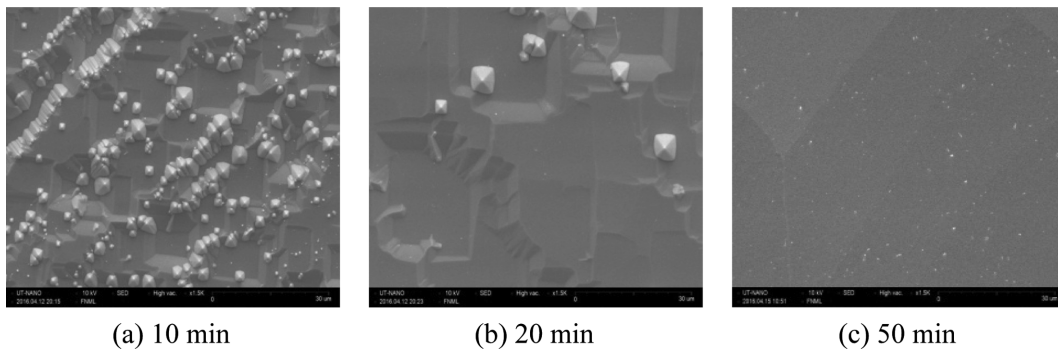


Fig. 5. SEM images of the surface of diamond wire-sawn single crystalline silicon wafers after pretreatment using TMAH for different times. (a) 10 min (b) 20 min and (c) 50 min.

Table 1
Light reflectivity of diamond wire-sawn single crystalline silicon wafers in the wave length range of 400~1000 nm

	Before Texturing	After Texturing
As-cut wafer	28 %	12 %
TMAH pretreated wafer	10 %	11 %

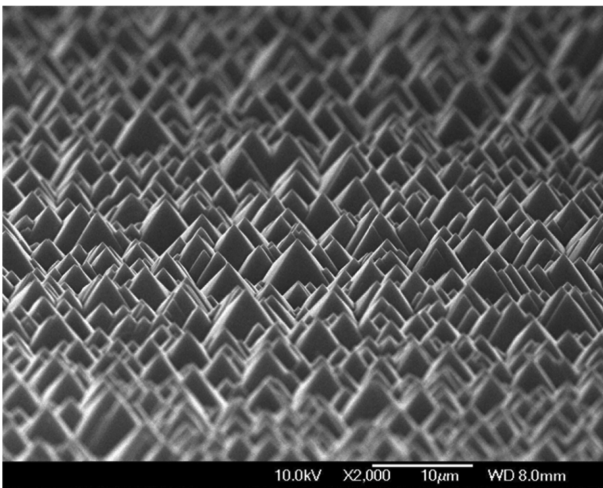


Fig. 6. SEM image of textured diamond wire-sawn single crystalline silicon wafer with 50 min pretreatment.

after TMAH pretreatment for 50 min. A SEM image of textured diamond wire-sawn single crystalline silicon wafer with pretreatment is shown in Fig. 6. We can see the pyramidal microstructure on the surface.

4. Conclusion

The promising method for solving the saw marks problem of diamond wire-sawn single crystalline silicon wafers was proposed and studied. A pretreatment step using TMAH was proposed and conventional texturing process with KOH solution after proposed pretreatment

was studied. The saw marks on the surface of the diamond wire-sawn silicon wafers could be effectively removed after pretreatment and KOH texturing steps, and they were invisible to naked eyes completely. Compared with the conventional KOH texturing process, the saw marks, uniformity of pyramidal structures, light reflectivity of the wafer surface were improved.

Acknowledgment

This work was supported by the research support program of the Dongyang Mirae University in 2015.

References

- [1] H. Wu, "Wire sawing technology: A state-of-the-art review", *Precision Eng.* 43 (2016) 1.
- [2] (<http://www.rockwellautomation.com/global/solutions-services/oem/application-profiles.page>).
- [3] K.H. Lee, "A study on the surface characteristics of diamond wire-sawn silicon wafer for photovoltaic application", *J. Korean Cryst. Growth Cryst. Technol.* 21 (2011) 225.
- [4] K.H. Lee, "Quality evaluation of diamond wire-sawn gallium-doped silicon wafers", *J. Korean Cryst. Growth Cryst. Technol.* 23 (2013) 119.
- [5] W. Li and D. Tsai, "Automatic saw-mark detection in multicrystalline solar wafer images", *Sol. Energy Mater. Sol. Cells* 95 (2011) 2206.
- [6] M. Lippold, F. Buchholz, C. Gondek, F. Honeit, E. Weffringhaus and E. Kroke, "Texturing of SiC-slurry and diamond wire sawn silicon wafers by HF-HNO₃-H₂SO₄ mixtures", *Sol. Energy Mater. Sol. Cells* 127 (2014) 104.
- [7] H. Takato, I. Sakata, K. Mase, S. Ishibashi, T. Harada, Y. Kondo and H. Asai, "Method for fabricating substrate for solar cell and solar cell", US Patent 20130306148 A1, Nov. (2013).
- [8] X. Yu, P. Wang, X. Li and D. Yang, "Thin Czochralski silicon solar cells based on diamond wire sawing technology", *Sol. Energy Mater. Sol. Cells* 98 (2012) 337.