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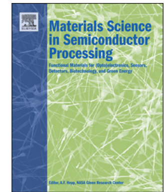

Comparison of diamond wire cut and silicon carbide slurry processed silicon wafer surfaces after acidic texturisation

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ABSTRACT

Our work focuses on the acidic etching of silicon wafers, cut via diamond wire (DW) or silicon carbide slurry process (SP). The DW and SP *as-cut* wafer surface structures have a significant impact on the evolution of the two resultant and different etched morphologies. The time-dependent development of the surface morphology for mono- and multicrystalline wafers is compared and analyzed via etch rates, reflectivity measurements and confocal microscopy. The *as-cut* structure of the differently sawn wafers defines a template where the etch attack preferentially occurs and predetermines the texturisation of the etched surface. Based on the experimental results it is possible to lower the reflectivity of the SP-sawn wafers by varying the acidic mixture. On the contrary, the DW-sawn wafers obtain only a small enlargement of the folded surface area during acidic texturisation and no influence of different acidic etch solutions on the reflectivity values was found. To create homogeneously texturized DW-sawn wafers of low reflectivity, an adaptation of the sawing process as well as the development of new etchants and new etch conditions is necessary.

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1. Introduction

The most widely-used and well-established technique to cut silicon bricks into thin wafers is sawing with a steel wire, SiC particle (slurry) as an abrasive and polyethylene glycol as coolant. The basic mechanisms of the slurry technique were investigated by Moller et al. [1]. They revealed the correlation of wafer surface roughness with the average indentation depth of the grains and the size distribution of the slurry particles. Further development led to the introduction of an ultrathin structured steel wire by Applied Materials for a more efficient transport of the SiC particles and therefore a faster cutting speed (25–35% higher productivity to the slurry process) [2]. More than 10 years ago, a steel wire patched with small diamonds, originally used for sawing sapphire, was utilized for silicon blocks with little success. Meanwhile the process, as well as the architecture of the wire, was adapted to the silicon material and now it is the most promising technique used to cut silicon blocks and bricks for the solar industry [3,4]. Compared to the widely-used slurry process, the advantages of the diamond-wire sawing technology yield higher productivity, less total thickness variation in the resulting wafer, a longer wire lifetime and therefore less downtime of the saw during wire changes. Another benefit is the possibility of kerf recycling due to the fact that there is no loose abrasive used and the coolant is only water [5]. Two disadvantages prevent the industrial breakthrough so far: one is the high cost of the diamond wire (over $200/km compared to $1/km for the slurry wire) and the other reason is the difficulty to cut multi-crystalline ingots, as the material leads to increased breakage rates during subsequent solar cell processing [6,7]. Further development led to the introduction of a fixed abrasive wire where

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the diamonds get attached with resin instead of electroplated copper alloy, resulting in less expensive diamond wires [3]. However, each sawing process leads to wafers with very different surface structures.

Wafers with such a damaged surface do not have advantageous semiconductor properties. The acidic etching removes the saw damage from the *as-cut* wafer surface and generates a certain surface morphology (texture) on the bulk silicon material consisting of shallow valleys and molds (similar to partly hemispherical structures) in the micrometer range. This texture has the function of reflecting the incident light to other spots within the wafer surface in order to increase the light harvest and therefore the efficiency of the solar cell.

Etching in photovoltaic industry (PV) is performed in the so called “feed-and-bleed” regime. Nitric acid (HNO_3) and hydrofluoric acid (HF) are continuously dosed into the etch bath while a consumed etch solution mainly consisting of HNO_3, HF and hexafluorosilicic acid (H_2SiF_6) is continuously removed from the etch bath. This regime should guarantee an almost stationary concentration of HF, HNO_3 and H_2SiF_6 as well as for reactive species (e.g. NO^+, $[N_4O_6]^{2+}$ [8]). However, to ensure a constant etch quality of 10,000 wafers, a more strictly analytical process control is required [9–14] to keep the concentration of the most prominent etch bath constituents in a sharp range.

The present work deals with mono- and multi-crystalline slurry and electroplated diamond-wire sawn wafers and examines the time-dependent development of the surface morphology (texture) of DW- and SP-sawn wafers by horizontal acidic etching. Etching mono- and multi-crystalline wafers with the acidic mixture of HF/HNO_3 was chosen for scientific reasons in order to determine the possible influence of the saw damage to the crystallinity of the wafers. This study is the continuation and conclusion of earlier published work [15,16].

2. Materials and methods

Industrially relevant acidic etching mixtures (mixtures with a much higher amount of HNO_3 than HF [17]) were prepared from hydrofluoric acid (40% (w/w)), nitric acid (65% (w/w) both from Merck Darmstadt, Germany) and hexafluorosilicic acid (35% (w/w) from Alfa Aeser, Karlsruhe, Germany). The composition of an etch mixture is denoted in % (w/w) in the order HNO_3/HF/H_2SiF_6. For example, an etch mixture consisting of 30% (w/w) HNO_3, 7% (w/w) HF and 20% (w/w) H_2SiF_6 is abbreviated as 30/7/20. Four wafer types were employed which differ in the sawing method as well as the crystallinity (mono–SP, multi–SP, mono–DW, multi–DW). 20 neighboring wafers from one cut of one brick were taken and each wafer ($156 \times 156\ mm^2$) was divided via laser cutting into 100 $15 \times 15\ mm^2$ (with 3 mm residual on each side) small wafer pieces. From the center area of each $156 \times 156\ mm^2$ wafer a stack of 10 neighboring wafer pieces were chosen for one etch experiment. Mono-crystalline Si ⟨100⟩ and neighboring multi-crystalline silicon wafer pieces with $15 \times 15\ mm^2$ diameter and 180–200 μm thickness were etched horizontally and simultaneously with varying time steps from 10 to 450 s at 10 °C (Peter Huber Kältemaschinenbau GmbH Offenburg, refrigerated bath K12+CC2). The wafers were hooked in forceps and were held in the middle position of 50 mL etch solution. After etching for a specific time step, the wafers were rinsed in water and dried. Ahead of the surface evaluation the wafers were dipped into 2.8% (w/w) NaOH solution in order to clean the wafer surface from eventually-generated porous silicon. To follow the development of the etched surfaces, the reflectivity was measured at 700 nm (Specord 250plus with integrating sphere from AnalytikJena AG) and confocal microscopic measurements (Olympus LEXT OLS4000 from Olympus Deutschland GmbH, Hamburg) were performed. The reflectivity measurements were taken at one reading point at the center of each wafer surface with a spot size of 3–5 mm². Since the orientation of the DW wafer matters, all surements were conducted in horizontal orientation of the rift structures [18]. With the help of the confocal measurements the *S*/*P* values were calculated. *S*/*P* corresponds to the ratio of the total folded surface area consisting of all hills, rifts and valleys to the geometric (top-view projected, $15 \times 15\ mm^2$) area. The wafers were weighed before ($m_{as\ cut}$) and after (m_{etched}) etching with 0.1 mg accuracy to calculate the mass of etched silicon, Δm, according to $\Delta m = m_{as\ cut} - m_{etched}$. From Δm, the density of silicon (ς_{Si}), and the area of the etched wafer piece (A_{wafer}), the total thickness loss (the sum for both wafer sides), Δd, is obtained by $\Delta d = \Delta m / (\varsigma_{Si} A_{wafer})$. To calculate the etch rate, *r*, given as $r = \Delta d / t_{etch}$, the time of etching, t_{etch}, resulting from the difference of the immersion time (t_{imm}) and the time of the induction period, t_{ind}, is given by $t_{etch} = t_{imm} - t_{ind}$. The induction period characterizes the initial time of immersion which passes without any visible reaction (usually between 1 and 3 s). The explicit consideration of the induction period is necessary to calculate the true etch rate; however, it is of less importance for the industrial process.

It turned out to be advantageous to use the change in reflectivity, ΔR, calculated from the absolute reflectivity of the wafer after etching, R_{etched}, and the absolute reflectivity of the same wafer before etching, $R_{as\text{-}cut}$, according to $\Delta R = R_{etched} - R_{as-cut}$. This procedure reduces the impact of the studied wafers' local-surface heterogeneity so that tendencies within the etch series become clear.

3. Results and discussion

3.1. As-cut wafer surface

The visual inspection of the two differently sawn wafer types (SP and DW) reveals their differences in the *as-cut* structure. The SP wafer has a regular, gray appearance in contrast to the DW wafer with a silver, shiny surface and clearly visible parallel grooves. The slurry-sawn (SP) surface is characterized by a homogeneously distributed rough, dentate, fractured surface (Fig. 1). In contrast, the diamond-wire-sawn (DW) wafer features smooth parts, parallel rifts, individual fractures and areas of cracks along the rifts (Fig. 2). The initial reflectance of the DW wafer with $R_{as\text{-}cut} = 26\%$ is typically around 2% higher than for the SP wafer with $R_{as\text{-}cut} = 24\%$. The smooth regions of the DW-sawn wafer might be considered as the reason for these higher reflectivity values.

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