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Dislocation formation in seed crystals induced by feedstock indentation during growth of quasimono crystalline silicon ingots

M. Trempa [a,*], M. Beier [a], C. Reimann [a,b], K. Roßhirth [a], J. Friedrich [a,b], C. Löbel [c], L. Sylla [c], T. Richter [c]

[a] Fraunhofer IISB, Schottkystr. 10, 91058 Erlangen, Germany
[b] Fraunhofer THM, Am St.-Niclas-Schacht 13, 09599 Freiberg, Germany
[c] SolarWorld Innovations GmbH, Berthelsdorfer Str. 111A, 09599 Freiberg, Germany

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ABSTRACT

In this work the dislocation formation in the seed crystal induced by feedstock indentation during the growth of quasimono (QM) silicon ingots for photovoltaic application was investigated. It could be shown by special laboratory indentation experiments that the formed dislocations propagate up to several millimeters deep into the volume of the seed crystal in dependence on the applied pressure of the feedstock particles on the surface of the seed crystal. Further, it was demonstrated that these dislocations if they were not back-melted during the seeding process grow further into the silicon ingot and drastically reduce its material quality. An estimation of the apparent pressure values in a G5 industrial crucible/feedstock setup reveals that the indentation phenomenon is a critical issue for the industrial production of QM silicon ingots. Therefore, some approaches to avoid/reduce the indentation events were tested with the result, that the most promising solution should be the usage of suitable feedstock particles as coverage of the seed.

1. Introduction

In recent years the quasimono (QM) directional solidification technology was developed to produce high quality mono-crystalline silicon ingots for solar cell application [1]. However, so far it is not clear, whether the QM technology can play a significant role in the future photovoltaic world market, because it has still to overcome several challenges to be competitive to the well-established Czochralski method for the growth of single crystals. Prominent tasks are e.g. the metal contamination of the seed crystals resulting in larger areas of lower lifetime in the bottom region of the QM ingots [2,3], the multi-crystalline grain growth in the border regions of the ingots [4], and the dislocation formation at the seed joints [5,6]. All these phenomena can drastically reduce the ingot yield of high quality mono-crystalline wafers resulting in higher production costs. Furthermore, several problems concerning the dislocation formation in the seed volume have not been solved yet. These dislocations can propagate from the seed crystal into the QM silicon ingot during the growth and cooling processes. Different factors like surface damage of the seed or contact points between two seeds were made responsible for this dislocation formation in the literature [7]. One further critical point could be the indentation of the upper seed crystal surface by feedstock chunks/chips [8]. This indentation leads to a mechanical deformation of the seed crystal at the surface, inducing dislocations. The generated dislocations can propagate into the volume of the seed crystal during heating up, multiply and remain in the seed as long as they will be not molten back during the seeding process. The remaining dislocations can then propagate into the silicon ingot during crystal growth and ingot cooling phase and can therefore drastically reduce its material quality. However, so far a detailed study is missing on the effect of the indentation of the seed crystal. Especially it is not clear how far the defects generated by indentation would propagate into the seed volume in function of the used time-temperature profile and the silicon feedstock load. Once the effect of the indentation on the seed crystal is known, technological solutions can be proposed to overcome the indentation problem for the industrial application. Therefore, in this work the feedstock indentation phenomenon was systematically investigated by so called "indentation experiments" in a laboratory setup. Thereby, the following questions will be answered:

- Which pressure (ratio of load to contact area) of the indenters is necessary to initiate dislocations at the seed crystal surface?
- How does the dislocation penetration depth in the seed crystals

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[*] Corresponding author.
E-mail address: matthias.trempa@iisb.fraunhofer.de (M. Trempa).

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depend on the occurring pressure values, temperature and time scales, and which process conditions lead to a critical dislocation penetration depth for industrial processes?

- Are there any practical approaches to reduce the propagation of the dislocations into the silicon seeds in such a way that the dislocation structures could be reliably back-melted during the seeding process and therefore the indentation process becomes harmless?

2. Experimental setup and characterization

2.1. Indentation experiments

The schematic setup of the indentation experiments is sketched in Fig. 1a. At first three silicon indenters in terms of grinded four-sided pyramids with an edge length of 1 cm were bonded on a monocrystalline silicon wafer to give them a good mechanical stability against twisting and tilting. Beneath the pyramids a mirror polished [110] oriented single crystalline Si seed (Cz material) with a typical thickness of 20 mm is placed. On top of the silicon wafer with the silicon pyramids, graphite loads with different geometries and masses were placed to simulate the weight of the silicon feedstock material as it is used in standard crystallization experiments.

The pressure which the indenters impinge on the seed crystal surface is defined by the ratio of the mechanical load of the graphite and the tip area of the silicon pyramid which is in contact with the seed crystal surface.

The four pyramidal side areas were manually grinded to create an as small as possible tip area of the pyramids ($<1\ \mathrm{mm}^2$). However, the final tip area is a consequence of the accuracy of the manual preparation process and leads therefore to a slightly spread distribution of its values. Furthermore, the exact determination of the tip area which was done by light microscopy (LM) is not trivial due to reflection effects of the shiny silicon surface. Therefore, the minimum and maximum of the tip area which could be theoretically in contact with the seed crystal surface was measured on the LM images as shown in Fig. 2. Based on these values, the resulting maximum and minimum pressures and finally the mean pressure of the indenters were calculated. The error E which is made by this method was defined to be $E = \mathrm{pressure}_{\mathrm{max}} - \mathrm{pressure}_{\mathrm{mean}}$.

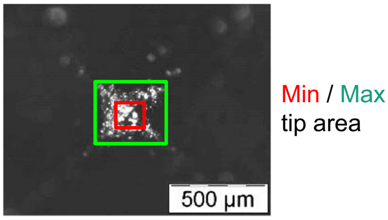

Fig. 2. LM image of a silicon pyramid tip as used for the indentation experiments. Marked is the maximum and minimum tip area which was taken to calculate a mean pressure value for each indenter.

To realize a wide range of mechanical load and in consequence of pressure two furnaces with different scales were used. On the one hand small graphite cylinders with moderate weights of 0.8 kg and 1.5 kg weight were put on cylindrical silicon seeds (10 cm in diameter) within a G0 crystallization furnace which is normally used to grow cylindrical ingots with 10 cm in diameter and an ingot weight of 1.2 kg [9]. On the other hand large graphite blocks with even higher weights of 9.9 kg and 19.4 kg were put on rectangular silicon seeds ($\sim 100 \times 200\ \mathrm{mm}^2$) within an annealing furnace which allows to anneal e.g. silicon-ingots up to an edge length of 22 cm and an ingot weight of 15 kg (G1 scale).

The temperature profiles used for the indentation experiments were close to the process conditions during a typical G0 or G1 crystallization process. The maximum temperature was set below the melting point (1350–1390 °C) to prevent the melting of the silicon pieces which was controlled by an additional thermocouple close to the seed crystal surface (see setup in Fig. 1a).

Additionally the annealing time (at maximal temperature) was varied between 16 h and 40 h to simulate typical crystallization runs respectively in laboratory and industry.

In Table 1 all parameters for the indenter experiments are listed namely the graphite weight, the resulting load on each silicon pyramid, the range of the mean tip areas within one experiment, the resulting range of mean pressure values which the indenters impinge the seed crystal surface, the seed crystal temperature and the annealing time.

After the temperature treatment the silicon seed crystals were cut in vertical direction along the (100) plane through the visible

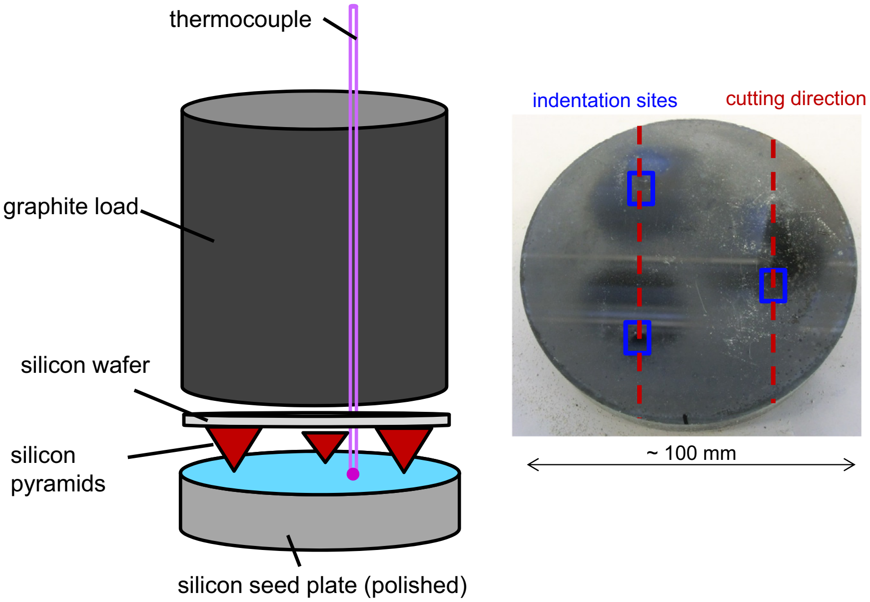

Fig. 1. Schematic setup of the indentation experiments (a) and top-view of a seed crystal after temperature treatment including marks of indentation sites (blue squares) and cutting directions (red dashed lines) (b). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).