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Dislocation formation in seeds for quasi-monocrystalline silicon for solar cells

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Abstract

An investigation of two industrially cast quasi-monocrystalline silicon blocks revealed a high dislocation density originating at intersections between the seed crystals. This may be ascribed to three different generation mechanisms. Firstly, a dislocation cell structure was observed in the seed crystals, probably as an effect of poor surface preparation of the seeds. Furthermore, clusters of dislocations form around contact points in the interface between two neighbouring seeds. At contact points, the two monocrystalline silicon seeds plastically deform and sinter together. Dislocation rosettes form as a result of an indentation mechanism at high temperatures. A third mechanism acts at the bottom surface, where dislocation clusters also form by indentation of contact points between the seed and the crucible. Since dislocations forming in the seeds will continue into the growing ingot, it is crucial to depress the dislocation formation in the seeds.

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1. Introduction

Growth of quasi-monocrystalline (quasi-mc) silicon by directional solidification is a relatively new technique for growing industrial single crystalline silicon [1,2], although the principles of using crystalline seed crystals, during directionally solidification, to obtain a specific direction or a desired grain boundary are well established [3]. Since slices of single-crystalline Czochralski (CZ) single silicon crystals are used as seeds, limitations on the seed size make it necessary to use several seeds arranged along the crucible bottom, also called "split seeds" [4]. Early in the process, the top part of the seed is carefully melted together with the feedstock material placed above, and as solidification proceeds the growing crystal will obtain the same orientation as the seeds in the bottom, which is a $\langle 100 \rangle$ normal parallel to the growth direction in most cases. Important challenges are still preventing the technique from fulfilling its potential, and some of the difficulties are related to crystal-defect formation during crystallization and cooling. Defects such as dislocations, sub-grain boundaries (sub-GB) and stacking faults are created at the interface between neighbouring seeds, as well as near the edges of the crucible. Observations of the dislocations arranged in cascades, increasing in density towards the top of the block [5,6].

A portion of the seed crystals, which are solid throughout the process, will have temperatures close to the melting temperature of silicon. The seed crystals and the interface between the parallel neighbouring seeds are therefore important regions to study. Dislocations or sub-grain boundaries created or existing here have the potential to continue into

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the growing material at the high temperatures that exist during the process. In the present work, dislocation structures in the seed region have been investigated in detail.

2. Experimental

2.1. Sample preparation

Two neighbouring blocks from an industrially cast quasi-monocrystalline ingot have been investigated; Fig. 1a shows a sketch of the two, originally being part of an ingot cut into 16 blocks. The blocks were divided into four smaller parts, numbered 1–4, and each of the new blocks were cut into 10 mm thick slabs on the vertical side as illustrated on block 1, part 4. In addition, horizontal samples were cut. The seed interface separating two seed crystals in the bottom is located in the lower right corner in the figure showing block 1. The region of focus in this work is indicated in Fig. 1b, which shows a sketch of the two seeds, and indicates the various surfaces which were investigated.

Samples were cut with a tile saw, ground and polished on both sides for stress measurements. For defect characterization, the samples were etched in Sopori etch [7] (a 2:15:36 mixture of nitric, acetic and hydrofluoric acids) for 40 s and dipped in a 9:0:1 solution directly afterwards to avoid staining.

2.2. Infrared birefringence imaging

Infrared birefringence imaging (IBI) is a technique for investigating residual stresses in birefringent samples [8,9]. The IBI technique is based on the photoelastic effect, i.e., some materials, including silicon, exhibit birefringence that varies linearly with stress. In this work, a grey-field polariscope developed by Stress Photonics Inc. was used to quantify in-plane shear stress. A sample is impinged by circularly polarized light, i.e., two perpendicularly polarized plane waves with a quarter-wavelength $(\lambda/4)$ phase shift between the two waves. A birefringent material will introduce a slight phase difference $(\Delta \lambda)$ between the two perpendicularly polarized plane waves, as each of the two plane waves travel at different speed through the material. The light exiting the material will have a phase difference of $\lambda/4 + \Delta\lambda$, and consequently, the light will be elliptically polarized. A rotating polarizing filter measures the ellipticity of the light transmitted through the sample. By a series of trigonometric calculations, the ellipticity of the light relates to the phase difference $\Delta \lambda$. This value is directly proportional to the stress components in Mohr's circle, $(\sigma_x - \sigma_y)$, τ_{xy} and τ_{max} . A 320 × 256 InGaAs pixel array with a 1100 nm bandpass filter captures a two-dimensional map of stress across a silicon sample. In the IBI, we measure the difference in principal stress in the $0^{\circ}/90^{\circ}$ and $\pm 45^{\circ}$ orientations for $(\sigma_x - \sigma_y)$ and τ_{xy} , respectively. For a sketch of the setup and a detailed description of the technique see Ref. [8].

2.3. Defect characterization

For defect structure characterization, defect-etched samples were imaged by a light microscope (LM). A LVFESEM Zeiss Supra 55 VP scanning electron microscope (SEM) equipped with an electron backscatter diffraction (EBSD) detector was used for orientation measurements and grain boundary characterization.

3. Results

3.1. Overview of investigated regions

Fig. 2a is a mosaic of LM images showing the seed junction and the surrounding seed. The seed interface separating the two seed crystals is ~ 11 mm, and this region of the seed has been solid throughout the process. A junction is formed where the two seeds have melted together.



Fig. 1. (a) Sketch showing how two neighbouring blocks with dimension $250 \times 156 \times 156$ mm³ were cut. Block 1 contains a seed interface which lies very close to the edge. The white and blue regions illustrate the part of the seeds which is not melted, and the focus area is circled. (b) Sketch showing the two seed crystals forming the seed interface in block 1, and the different surfaces investigated is indicated with defined surface plane. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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