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Surface Examination of Structure Loss in N-Type Czochralski Silicon Ingots

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Abstract. In principle, growing a dislocation-free Czochralski silicon ingot is possible if the growth process is kept stable and below the critical resolved shear stress value. However, in practice, a considerable proportion of the Si ingots are remelted due to the generation of dislocations or the so-called structure loss. The assessment of the failed ingots is a crucial step toward higher yield. However, the characterization of Si ingots is challenging due to their high brittleness and the high concentration of dislocations related to slip. In this work, we develop a non-destructive method to investigate the ingots that have experienced structure loss and reveal the root causes of this failure. Many characteristic features have been found on the surface of Czochralski silicon ingots. Based on these features, the ingots are classified into seven major groups that could be related to the main causes of the structure loss. Furthermore, the temperature gradient of several ingots is revealed by careful measurements of the growth ridges' widths of these ingots. The results show that most of the failed ingots experience low-temperature gradients before the dislocation generation which agrees with the previous results. Three ingots have a clear particle hit on the surface, which caused an immediate transition to a multi-crystalline silicon structure. Particles are found on atomically smooth and rough interfaces, growth ridges, and surfaces in between. The surface examination method is a promising, fast, low-cost, and non-destructive technique that can be used to identify the most critical factors of structure loss in industrial ingots.

Keywords: Silicon, Czochralski, Defects, Surface-Examination

1. Introduction

Currently, more than 85% of silicon wafers have a mono-crystalline structure as this type has proven relatively higher efficiency and fewer defects compared to multi-crystalline silicon wafers. Industrially, monocrystalline silicon wafers are cut from single-crystal silicon ingots that are grown by the Czochralski method [1]. The method, which was developed in 1931, has seen significant advancements in single-crystal growth over the past 50 years. The grown Cz ingots are known for their dislocation-free structure which was only possible because of Dash's practical procedure of dislocation elimination at the very early stage of the growth. At this time, the Cz is a highly sophisticated method that is controlled by several parameters. Understanding and improving the current method has become more complex due to additional layers of complexity. One of the crucial issues in the Cz-industry is losing the dislocation-free structure during growth, or what is so-called structure loss [2, 3]. This problem is reported to occur at different growth stages. Remelting the failed ingots is the only available solution in the industry, which ultimately decreases the production yield. To better study this phenomenon, we should first understand the mechanism and root causes of dislocation generation in the single silicon crystal, especially since growing dislocation-free Si ingots is entirely feasible as granted by Dash in 1958 [4]. The carrier lifetime, as well as mechanical stability of the silicon wafers, are dramatically affected by the existence of dislocations as has been reported by [5-7]. Due to the complexity of this process, many reasons can lead to structure loss. These factors can significantly vary based on the stage of growth, doping type, and hot zone design [2]. However, some studies reported that particles [8], thermal shock [4, 9, 10], and gas bubbles [11] are the main responsible ones for dislocation generation. However, this classification is wide and cannot improve the growth process as many factors can introduce particles and/or gas bubbles in the melt. Similarly, thermal shock is also a result of many actions that can be involved during growth. Although Czochralski Si growth is well-explained in some articles and book chapters [3, 12-15], the generation of dislocations was poorly clarified. Still, few articles are devoted to covering the critical issues of Cz-Si growth such as structure loss. This also referred to the fact that structure loss has not been studied systematically yet.

2. Experimental

This work is based on the investigation of forty n-type Czochralski Si ingots that experienced structure loss during growth. The phosphorus-doped ingots were supplied by industry partners. Hundreds of images were taken with a digital microscope Dino-lite AM7915MZT. Further analysis of the images was done by the image-J software. All the run data of the ingots were collected and analyzed to better correlate the surface features and the growth process in which statistical data analysis tools are used.

3. Results and Discussion

3.1 Major surface features during the body growth

The structure loss position is indicated by the disappearance of the growth ridges as the dislocated sites are more energetically preferable for Si growth than the atomically smooth interface of the growth ridges for silicon growth. Figure 1 shows an example of the growth ridge stop due to the structure loss in an industrial Cz-ingot. The dislocations were traced on the ingot’s surfaces by the meaning of the slip lines as shown in Figure 1. Two types of dislocations are found: (i) growth dislocations: generated at the solid-liquid front and located at the growth ridge stop, (ii) post-growth dislocations: generated behind the solidification front and revealed on the surface by feather-like slip lines as shown in Figure 1 b). The growth ridges are distinct features that can reveal valuable insights about growth conditions. Three major shapes of growth ridges are found during the careful examination of the Cz-ingots as can be seen in Figure 1. The majority of the failed ingots exhibited flat and wide growth ridges, as depicted in Figure 2. a. Moreover, the presence of distinctly flat and wide ridges, classified as type-c in Figure 2, suggests a low temperature gradient or what is commonly referred to as "cold melt" in the industry.

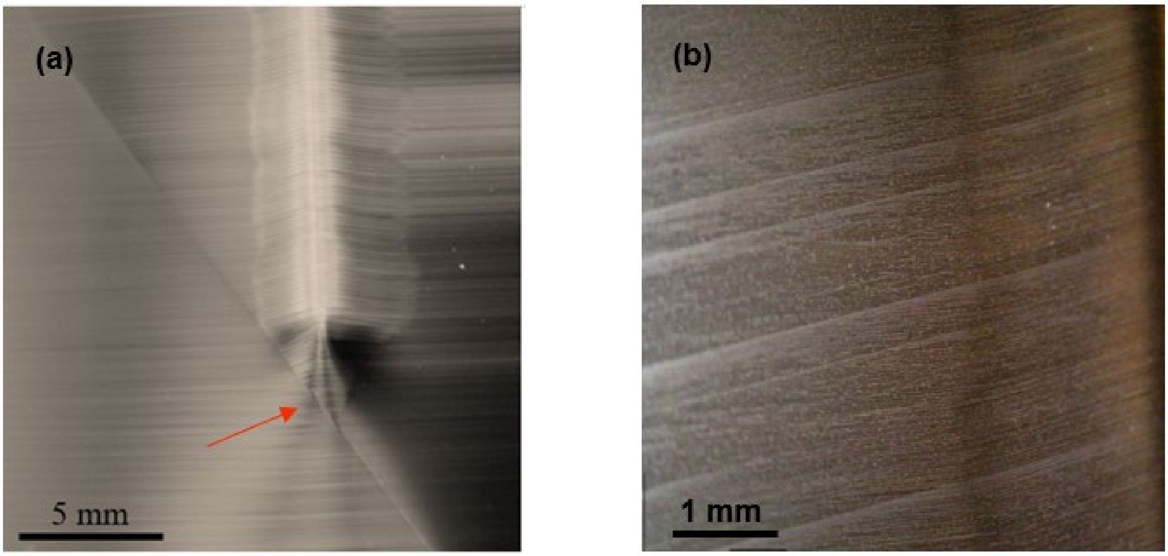

Figure 1. Growth ridges of structure loss n-type ingot (diameter 22''): a) growth ridge disappearance, indicated by the red arrow, due to the growth dislocation, b) feather-like slip lines appeared behind the solidification front.

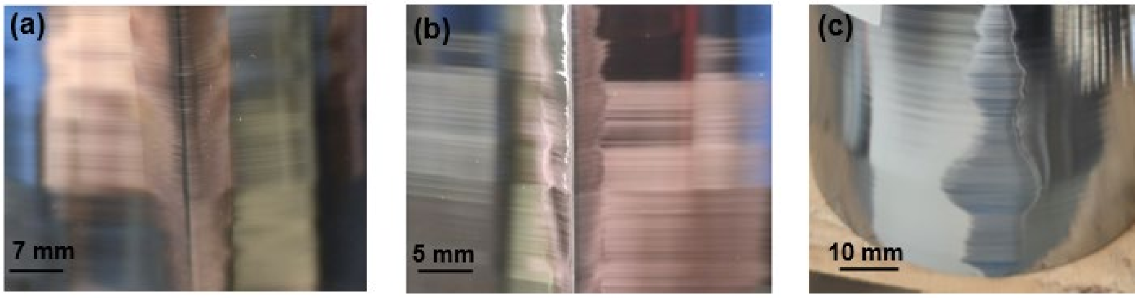

Figure 2. Major shapes of growth ridges are found by the surface examination of Cz-Si. (a) wide a flat growth ridge with no clear edges. (b) narrow growth ridge with well-defined edges. (c) very flat and wide growth ridge.

A thorough examination of the growth ridge widths shows that about a quarter of the studied ingots experienced a steep change in the growth ridge width by 40-50 % as can be seen in Figure 3. These severe fluctuations in the growth ridges' width reflect temperature fluctuations as also reported by Stockmeier et al. [4]. This is further supported by the analysis of process data associated with these ingots, which confirms the presence of temperature fluctuations during the solidification process, particularly, a sudden drop in the melt temperature. Figure 4 shows an obvious particle hit at the facet region during the growth, which caused an immediate transition from mono- to the multi-crystalline structure. The same case was reported in the other two ingots. Notably, the distance between the growth dislocation and the transition to multi-crystalline silicon (Mc-Si) varies among ingots and can be categorized as either short or long distance. Some ingots that exhibit a rapid transition to the Mc-Si structure may also contain hidden particles that are not visible on the surface. Additionally, it has been observed that certain structure loss ingots exhibit significant diameter variations, which may indicate fluctuations in the pull speed during the body growth process as illustrated in Figure 4. b.

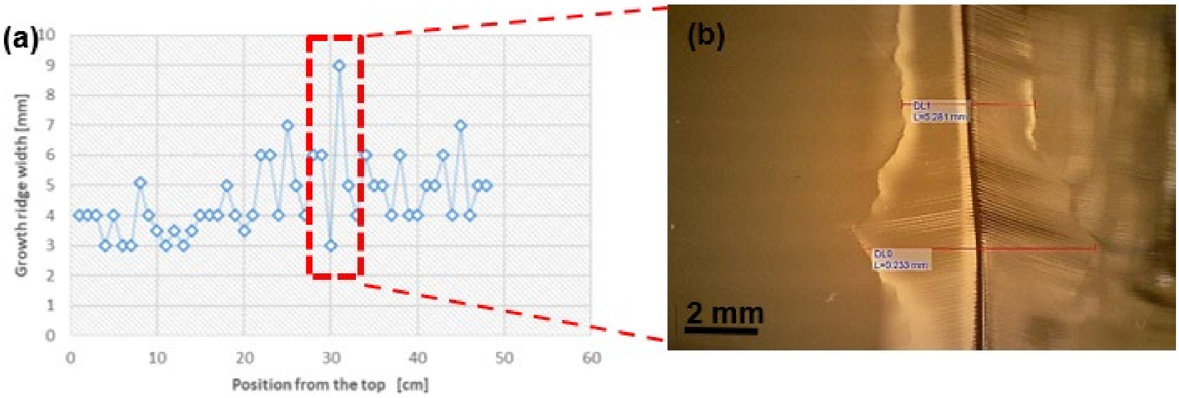

Figure 3. Variation in growth ridge width for a structure loss ingot. a) Growth ridge width as a function of the ingot position, b) optical micrograph of the selected area in the same ingot.

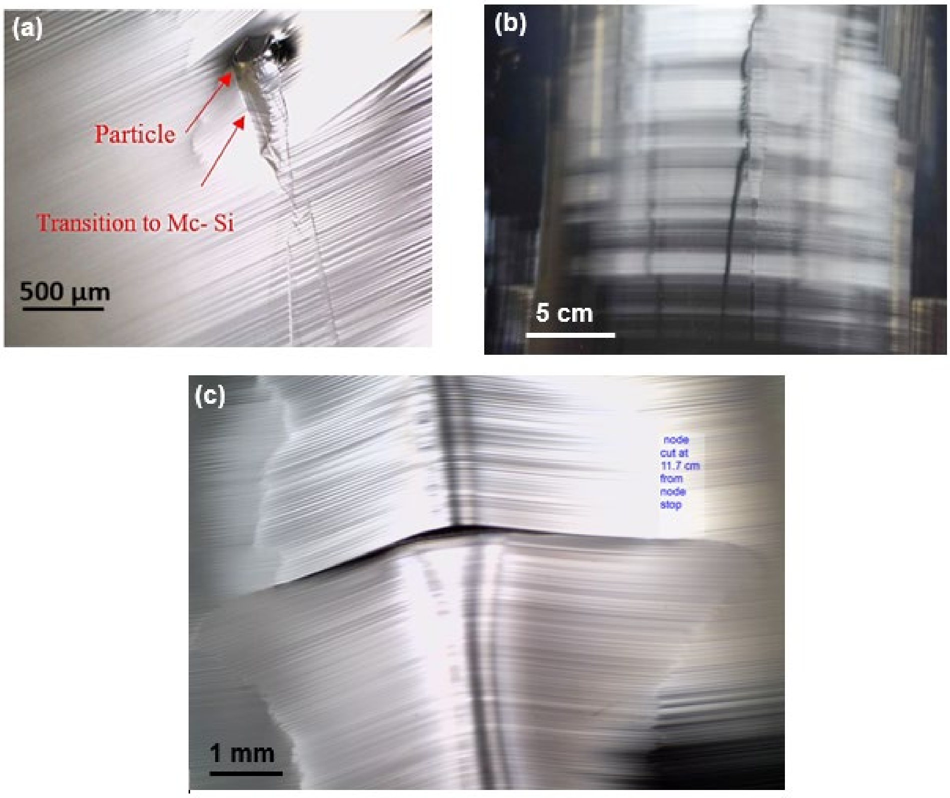

Figure 4. (a) Particle hit at the growth ridge causes an immediate termination of the ridges and nucleation of new Si crystals. (b) Diameter fluctuations in structure loss ingot. (c) A notch in the growth ridge appeared at 11.7 cm before the growth ridge stop.

3.2 Major surface features during top-cone growth

During the investigation of structure loss in the top cones, different shapes were observed, including flat and acute top cones as shown in Figure 5. Most of the SL- ingots exhibited flat top cones, while fluctuations in facet widths were evident in all top cones, indicating high-temperature fluctuations. Additionally, asymmetry of facets was noticed, which may be related to crystal rotation during the growth process.

3.3 Basic categories

Building on the previous observations made on structure loss ingots, it is possible to classify the defective ingots into major categories based on the identified characteristics and features. The major surface categories observed in the investigation of structure loss ingots are as follows:

1. Steep change in the ridge width: Some ingots showed a sudden and significant change in the width of their growth ridges, which could suggest possible fluctuations in the melt temperature. This implies that there may have been rapid variations in the cooling rate or melt temperature during the growth process of these ingots.
2. Flat and wide growth ridge: Ingots that show flat and wide growth ridges, suggestive of a low-temperature gradient during growth.
3. Asymmetric growth ridges: Ingots that display uneven or asymmetric growth ridges, suggesting possible crystal rotation irregularities.
4. Obvious particle hit: Ingots with obvious particles on the surface, indicating potential contamination during the growth process. Contamination primarily originates from the dissolution of the crucible or the graphite lining of the puller.
5. Short distance to Mc-Si: Ingots that exhibit a short distance to the multi-crystalline silicon (Mc-Si) structure, can indicate a hidden particle that hit the solid-liquid interface and was not obvious on the surface.
6. Diameter fluctuation: The fluctuations in diameter can indicate potential variations in pull speed.
7. Notch in the growth ridge: The presence of this feature, see Figure 4 .c, was observed in certain ingots up to 8-10 cm before the growth ridge stop, potentially indicating crystal twinning as reported in a previous study that has suggested a possible association with gas bubbles or slow growth rate [16].

4. Conclusions

In conclusion, surface examination proves to be a valuable, quick, cost-effective, and non-destructive method for characterizing structure loss Cz-Si ingots. Through careful surface examination, several surface features were identified, leading to the establishment of categorization into seven major groups. This serves as a starting point for data analysis, as a strong correlation is found between the surface features and process data. Notably, around half of the cases of structure loss were found to be related to temperature instability, low melt temperature, and short stabilization time. By optimizing these parameters, production yield can potentially be increased.

Data availability statement

The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Author contributions

Rania Hendawi: investigation, methodology, and writing-original draft. Gaute Stokkan: conceptualization. Eivind J. Øvrelid: conceptualization, Marisa Di Sabatino: conceptualization, supervision and writing-review and editing.

Competing interests

The authors declare that they have no competing interests.

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