

Applicability of a Flat-Bed Birefringence Setup for the Determination of Threading Dislocations of Silicon Carbide Wafers

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Abstract. Screw-type dislocations like micropipes (MP) and threading screw dislocations (TSD) are prohibiting the function or at least diminishing the efficiency of electronic devices based on silicon carbide (SiC). Therefore, it is essential to characterize wafers in an efficient and fast manner. Molten potassium hydroxide (KOH) etching or white-beam X-ray topography (SWXRT) are either destructive in the case of KOH etching or not economically viable in the case of SWXRT for an in-depth characterization of every wafer of one SiC crystal. Birefringence microscopy is being utilized as a fast and non-destructive characterization method. Instead of microscopic setups, commercially available flat-bed scanners equipped with crossed polarizer foils can be used for fast large-area scans. This work investigates the feasibility of such a setup regarding the detection rate of MPs and TSDs. The results of a full-wafer mapping are compared with birefringence microscopy and KOH etching. In the investigated sample clusters of MPs caused by a polytype switch in the beginning of the growth could be identified by both birefringence microscopy and the flat-bed scanner setup, as well as small angle grain boundaries and threading edge dislocation (TED) arrays. However, the resolution of the scanner was not sufficient to identify TSDs. Nevertheless the setup proves to be an easy-to-setup and cheap characterization method, able to quickly identify defect clusters in 4H-SiC wafers.

Introduction

In recent years, the physical vapor transport (PVT) method to grown bulk silicon carbide (SiC) crystals has been established to grown large-area micropipe-free material for the usage in high performance power electronic devices. While micropipes (MP) have largely been eliminated from commercially available wafers, they still can occasionally emerge due to unintentional polytype switches or post-growth annealing treatment [1]. Although SiC crystals with an apparent polytype switch are generally discarded for processing, a polytype switch in the initial seeding phase from 4H to for example 15R and back to 4H will not be visible from the crystal's surface. Such a switch can happen due to suboptimal growth parameters [2]. The distinction between medium-grade and high-grade SiC wafers most often depends on the presence of MPs. Furthermore other defects like threading edge dislocations (TED) or threading screw dislocations (TSD) are still apparent in the currently available 4H-SiC wafers, diminishing the performance of devices [3]. White-beam X-ray topography (SWXRT) allows a non-destructive and highly detailed investigation of dislocations in SiC [4], however it is also expensive considering the beam-time required for full-wafer mappings,

in addition to a limited availability of synchrotron sites [5]. In recent years, more compact systems like the Rigaku XRTmicron tool were developed, bridging the gap between SWXRT and birefringence microscopy in availability and setup dimensions. Molten potassium hydroxide (KOH) etching is another accessible characterization method, enabling the determination of dislocation density and types in a timely and cheap manner [6]. However, after the etching process the sample requires further polishing to remove the etch pits, resulting in a loss of bulk SiC material. From an economical point of view this makes this method undesirable. Birefringence imaging however can be used for quality control of grown crystals regarding micropipes as a cheap and non-destructive method.

It has been shown that birefringence microscopy utilizing the rotating polarizer method allows to quantitatively investigate MPs and also TSDs, as long as the Burgers vector of the dislocation exhibits an edge component [7, 8]. Image processing techniques can be utilized for an automatic evaluation of the defect density regarding MPs and TSDs [9]. However with the rise of larger SiC wafers of up to 200 mm diameter, the required dimensions in a microscopic setup start to limit full-wafer mappings. In addition, due to the sequential nature of mapping with a microscope the required time to map larger wafers rises increasingly, up to a power of two.

In this work we investigate the applicability of a new fast-scanning birefringence setup and its limitations regarding the ability to detect the different defect types such as MPs and TSDs. For this, a 4H-SiC crystal was grown via the PVT method, sliced into wafers and polished. The wafers were examined with our new flat-bed scanning setup and compared with the results of the birefringence microscopy. To evaluate the detection rate of MPs and TSDs, the wafers were then etched with molten KOH.

Experimental

A 4H-SiC crystal was grown with the PVT method. The wafers were sliced and polished to allow an optical characterization. The wafers are cut 4° off-axis and exhibit a thickness of $1200\ \mu\text{m}$.

Flat-bed birefringence setup.

A commercially available flat-bed scanner (Epson perfection v800) with a maximum resolution of 6400 dpi and a scanning area of up to $254 \times 203\ \text{mm}$ was utilized. The SiC-wafer is placed between two linearly polarizing foils, placed in a 90° degree configuration in respect to each other so that passing light is fully extinguished. One scan of a 100 mm diameter sample takes approximately 7 minutes.

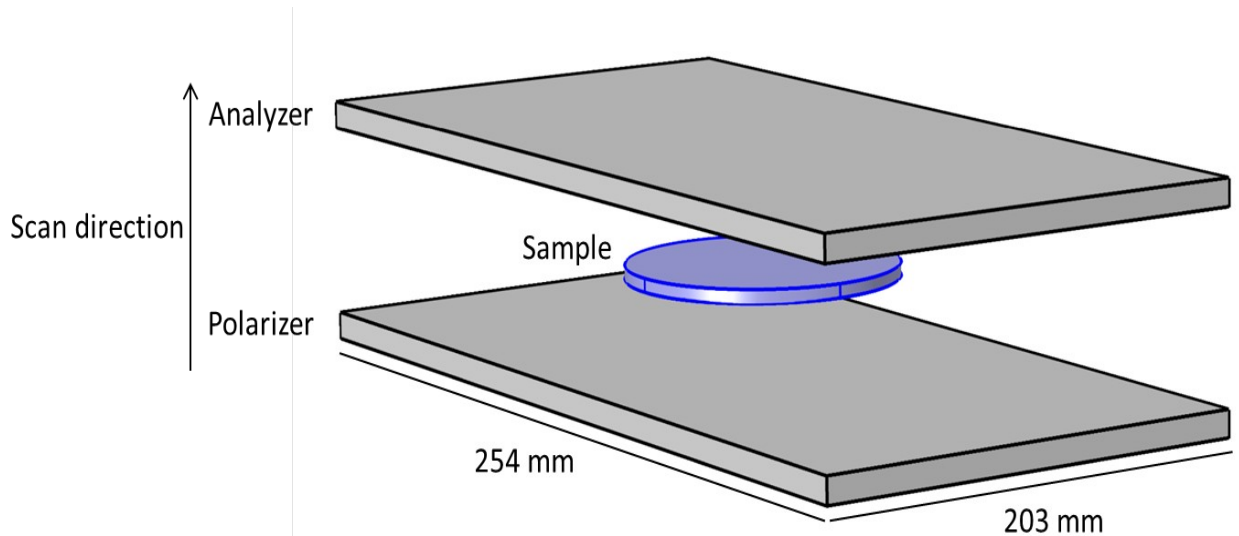


Figure 1: Schematic of the flat-bed scanner setup.

Polarization microscopy. For the birefringence microscopy a Zeiss Imager.Z1m microscope belonging to the Crystallography of the Albert-Ludwigs-University Freiburg was used. The sample was mapped with 5x magnification and the images stitched with the accompanied Zen software. Further image editing was done to remove the brightness shift of the respective images before stitching. One mapping took approximately 30 minutes. Due to the microscope's setup, it took 5 scans to completely map one 100 mm wafer.

KOH Etching. The KOH etching was carried out using the fully automated FAU KOH-etching setup, which is able to perform etchings of multiple 150 mm to 200 mm SiC wafers.

The samples were placed into a KOH melt of 520°C for minutes until the dislocations are etched sufficiently.



Figure 2: FAU KOH-etching setup

Discussion

Comparison between flat-bed scanner image and stitched microscopic image. Fig. 3a) depicts the result of the birefringence measurement done with the flat-bed scanner setup while Fig. 3b) shows the image taken by birefringence microscopy with the Imager.Z1m. In the latter case, a magnification of 200x was employed. The faint repetitive rectangular pattern is an artefact of the stitching process. Both setups are able to show a MP cluster in the center of the wafer. This cluster was caused by a polytype switch during the start of the crystal growth and is not visible in an optical scan. In addition, small angle grain boundaries (SAGB) can be observed at the edge of the wafer. These SAGBs also extend into the center of the wafer, which are apparent in the scan as well. In Fig. 4a) a repeating pattern is visible in our scanner setup. This pattern was verified in Fig. b) and c) as TED arrays by KOH etching.

Apart from that, the sample depicts a homogeneous intensity distribution in both images 3a) and 3b). The black dots visible are carbon inclusions caused by a process irregularity. In general, there are no apparent features missing in the birefringence image taken by the flat-bed scanner setup compared with the one taken by the Imager.Z1m microscope. The MP cluster in the center of the sample is magnified in Fig. 3c) and 3d), where 3c) belongs to the scanner setup and 3d) to the microscopic setup. For an easier comparison, two MPs are marked as 1 and 2. The MPs are clearly visible in both images, as well as a faint hexagonal pattern in the facet region. However, this pattern was not detected after the KOH-etching was completed. Since it is also apparent in the optical scan without birefringence, it is most likely a variation in nitrogen doping concentration in the facet, which was already reported in literature [10]. Overall, the result of the flat-bed scanner setup proves to be comparable with the one taken by birefringence microscopy.

Limitations of the flat-bed scanner setup. The limitations of this setup also have to be considered. Figure 3e) shows another magnified image of the MP cluster, concentrating on the MPs marked as 1 to 4. An additional etch pit is marked as 5. This defect is identified as a TSD via microscopy. The image was taken after the sample has been etched with molten KOH. While these four MPs are visible in the birefringent images, the TSD marked with 5 doesn't show any intensity variation in neither the images taken by the flat-bed scanner setup nor taken by the microscope. The defect count of TSDs and MPs in Fig. 3e) was approximated as 33 in total by means of image analysis using an algorithm developed by the Research Center Jülich Institute for Advanced Simulation. From these 33 threading dislocations, nine were identified as MPs. As a conclusion, the defect cluster can be detected in general while a calculation of the total defect density is not feasible with only birefringence imaging, since from nine MPs only four showed a contrast in the birefringence measurements. The defect cluster depicted in Fig. 3c) and d) exhibits 427 etch pits belonging to

TSDs and MPs. 68 of these defects show a contrast in the images taken with the flat-bed scanner setup, while the remaining 359 defects cannot be observed by the setup.

MP 1 was further investigated in Fig. 3f), where the focus of the microscope was shifted to highlight the path of the MP. As discussed in [7], the MP doesn't progress exclusively along the c-axis but also has an edge component. This behavior was observed for all MPs exhibiting an intensity shift in the birefringence measurements. Other MPs didn't show any influence on the detected strain in the crystal. It is calculated by Steeds and further covered by Ouisse et al. that screw dislocations without any edge component in the burgers vector are invisible in birefringence measurements, since they are photoelastically isotropic in the basal plane [8, 11]. Moreover, similar to the TSD marked with 5, we could not detect any TSDs featuring an intensity variation. While possible, it is highly unlikely that there are no mixed-type screw dislocations present in this sample. Therefore, the conclusion has to be that our flat-bed scanner setup doesn't feature the required dynamical range necessary for the detection of TSDs. It has to be noted that the Imager.Z1m microscope was not able to detect TSDs as well despite the fact that birefringence microscopy was able to be utilized for investigating TSDs in previous works.

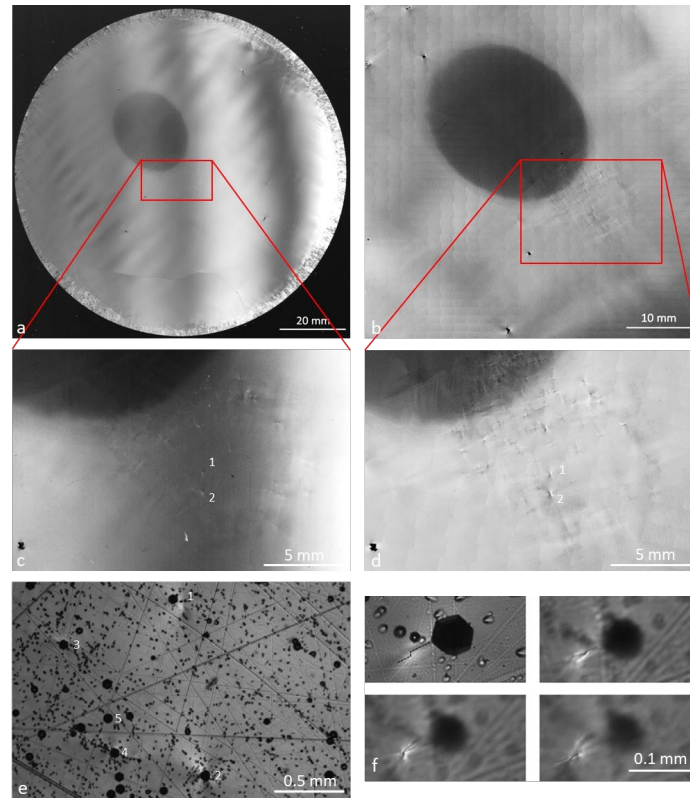


Figure 3: Birefringence mapping of the sample done with a) the flat-bed scanner setup and b) the Imager.Z1m microscope. c) and d) depict magnifications of the area marked with a rectangle in a) and b), respectively. e) pictures the region of the sample after KOH etching, marking MPs with birefringence signal as 1-4 and a TSD without any signal as 5. MPs 1 and 2 are the same as in the images c) and d). f) illustrates the path of MP 1 through the crystal. The path is marked with a dashed line.

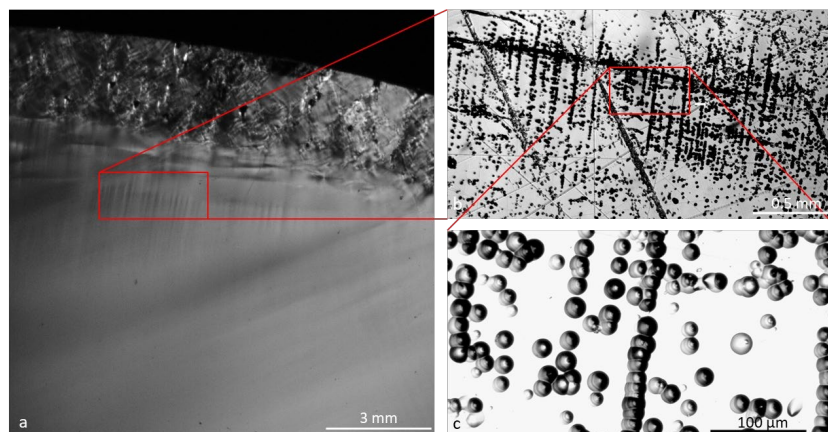


Figure 4a): Birefringence image of the sample before etching, taken with the flat-bed scanning setup, showing a TED array. b): Magnified image of the etched sample. c): Further magnification showing the single TEDs.

Summary

It has been shown that a partial amount of MPs could be identified by the flat-bed scanning setup equipped with two crossed polarizer foils. There was no notable loss in intensity compared with conventional birefringence microscopy. A MP cluster invisible in an optical scan could be detected as well as SAGBs and a TED array. KOH etching was done to investigate the sensitivity of the system to dislocations other than MPs. It has been shown that the setup was not able to detect TSDs or single TEDs. Nevertheless, due to its simple setup and since one full mapping of a 100 mm wafer only takes around 7 minutes, such a system could be an alternative as a non-destructive and fast characterization method to quickly identify SiC wafers with low or high MP density.

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