Characterization and Mapping of Crystal Defects in Silicon Carbide

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Abstract

A method is presented for detecting, counting and mapping dislocations and micropipes in n$^+$, undoped, and semi-insulating Silicon Carbide wafers. The technique is based on etching in molten Potassium Hydroxide (KOH). The polish-etch regime for sample preparation has been optimized to produce etch pits, which allow quick and accurate analysis of their optical contrast.

Etch pits from dislocations and micropipes are detected and differentiated by an image processing system that is sensitive to the optical reflection profile of the etch pit. The instrument probes an area of ≈ 800 x 600 μm^2 and is capable of producing topographic maps showing distribution of dislocation and micropipe densities (MPD). The MPD scan time for 2” wafers is less than two hours per wafer. The created MPD maps are in good agreement with the contrast images produced by the Synchrotron White Beam X-Ray Topography.

INTRODUCTION

Common defects in SiC are dislocations and micropipes. Micropipes are screw dislocations with Burger’s vectors of at least $2b_o$ in 6H-SiC and $3b_o$ in 4H-SiC where b_o is the Burger’s vector of a unit screw dislocation that is equal to the *c* lattice parameter (b_o = 15.18Å in 6H- and b_o = 10.12Å in 4H-SiC) [1]. These defects exist in the popular polytypes of SiC (6H, 4H, 15R and 3C), and their reduction in SiC substrates is the topic of current extensive research and development.

Several techniques used to reveal defects in SiC include molten salt etching, optical microscopy, synchrotron white beam X-ray topography (SWBXT), scanning electron microscopy (SEM), and atomic force microscopy (AFM). To date, there is no standard technique for mapping defects in SiC crystal, and most methods mentioned earlier are not practical for manufacturing purposes. In addition, some of the techniques mentioned above cannot count dislocation densities, which are known to affect the performance of SiC p-n junctions and Schottky barriers [2]. In addition, the optical methods have difficulty in discriminating micropipes and dislocations in etched semi-insulating SiC. The SWBXT system is widely considered to be an accurate technique for defect counting and can reveal both micropipes and dislocations. However, it is an expensive approach requiring access to a white beam source and is not economical for production.

We have developed an automated system based on KOH etching and optical contrast analysis of micropipes and dislocations in n$^+$, undoped, and SI SiC. KOH etching of SiC is easy, cost effective, and is a widely used technique for revealing defects in SiC. The system is comprised of an automated optical reflection microscope to acquire images of micropipes and dislocations and map their density and distribution. Image analysis software is used to discriminate between the various types of defects through a segmentation process. This process can be utilized for any semiconductor material that has visible defects on a scale down to ≈ 0.6μm. The technique provides several advantages. It is fast and accurate, making it ideal in a manufacturing environment; it can clearly distinguish micropipes from 1c dislocations in semi insulating SI, n$^+$, and undoped SiC samples; it can generate micropipe and dislocation maps, which are useful for analyzing growth uniformity in the growth process and to cross correlate with device yield during circuit manufacture.

Highlighted in this paper is an approach for monitoring the distribution and density of SiC defects utilizing KOH etching and optical microscopy, and a comparison with results from the SWBXT technique.

SAMPLE PREPARATION

Solid KOH pellets are heated above the melting point temperature (375^{o}C) in a nickel crucible. The SiC crystals are placed in nickel holders and etched at 400^{o}C - 550^{o}C for 10 – 20 minutes. Higher etching temperature will increase the removal of SiC [3]. Material removal is controlled via the etch rate, which depends on several factors, such as the availability of oxygen in the ambient around the system, the quantity of KOH used [3], the depth of damage, polytype differences as well as the orientation of the crystal.

Since the sample preparation is destructive, we use a wafer from the tail (top) end of the SiC crystal that is not less than 90% the nominal full diameter of the crystal’s standard wafers. The wafers are put through an etch-polish regime that will allow for precise defect analysis under optical microscopy. There are several etch-polish regimes that are applied to SiC samples. As-sawn wafers are etched to remove ~ 5-10μm. The C face will have a smooth polished surface, whilst the Si face reveals damage features caused by dislocation domain boundaries. The Si face is then polished to a roughness of ≈ 15 Å. The sample is then re-etched to remove ~ 0.5-1μm, after which it is ready for micropipe and dislocation analysis.

DEFECT CHARACTERIZATION

In order to detect, analyze and map micropipe and dislocation defects, an automated optical microscopy system was designed and implemented. The automated mapping system consists of an optical reflection microscope with a laser auto focus system, a charged coupled detector (CCD) camera with 1024 x 1024 pixel resolution, an automated stage, and a desktop computer that is interfaced with the system through graphics/programming software. The CCD camera, as well as the automated stage controller is interfaced with the computer. The graphics/programming package was also used to interface the stage controller and the CCD camera.

The optical microscope operates in bright and dark field modes. Bright field image is obtained by illuminating the object uniformly by incident light rays directed perpendicular to the sample surface. Light reflected back towards the objective is collected and focused into the eyepiece or camera to form a true color image at magnification up to 1250X. Surfaces that are perpendicular and reflective to the light rays appear bright. Objects, such as pits, scratches, and particles that scatter light, reflect less light back into the objective and appear dark.

In the dark field mode, the microscope works by enhancing the contrast of subtle topographical features. An occluding disk is placed in the light path, disrupting the direct vertical illumination as well as the light reflected from the object. Peripheral rays are reflected at oblique angles to the object plane. The result is bright reflectance only from ridges, pits, scratches, and particles. Reflective features that normally appear dark in bright field illumination are bright in dark field illumination.

Closed core dislocation etch pits, scratches, cracks and surface particles will appear as dark features in bright field and bright features in dark field. On the other hand, hollow core dislocations do not reflect light back in both modes. Micropipes, therefore, remain as dark features whether viewed in bright field or dark field mode, and since darkfield distinguishes between closed and hollow core dislocations this mode is most effective for discriminating micropipes and dislocations.

Etched wafers of various characteristics were studied to develop effective methods of defect image analysis. The simplest method for distinguishing the various defects utilizes the contrast of the etch features in the image. Etched micropipe pits appear hexagonal independent of the wafer doping level, whereas dislocation pits change appearance from round, in heavily doped n^+ SiC wafers, to hexagonal in undoped and semi-insulating SiC samples. As a result of their hollow nature, micropipes do not reflect much light and appear to be darker than other defects.

In heavily doped n^+ samples, discrimination of dislocations and micropipes is unambiguous due to their shape, size and contrast differences. An image of a section of the wafer is acquired under bright field microscopic setting. When counting only micropipes the acquired image is eroded to eliminate or minimize background noise and, uninteresting defects. The threshold level is set constant across the wafer surface, and the software is programmed to count the features (micropipes). Fig. 1 shows the result of a typical image analysis process for micropipes in a section of a heavily doped 6H-SiC wafer using the bright field mode of the microscope. Notice that Fig. 1a shows a normal microscope image that includes micropipe and dislocation defects as well as scratches from the mechanical polish process. The microscope is slightly defocused to distinguish such surface features like particles from subsurface damage and defects, and the image is captured at a higher light intensity (Fig. 1b). Applying an eroding filter results in Fig. 1c where micropipes are easily distinguished through segmentation.

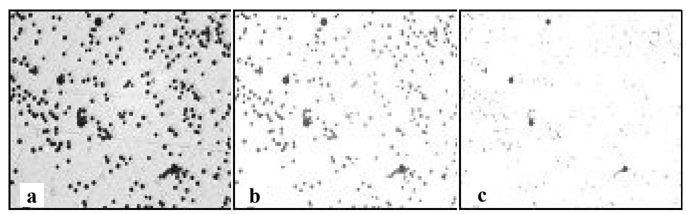

Fig. 1 an image frame of a 6H-SiC wafer with 100X magnification (a) with the same image taken at a higher exposure (b) and the result of erosion on the image (c) leaving micropipes visible for counting.

In undoped and SI SiC wafers, defect features of etched samples are not so easily distinguishable. In SI wafers, etched dislocations and micropipes are both hexagonal in shape and have similar lateral extent. However, these features can be segregated by utilizing the darkfield mode of the microscope. Fig. 2a shows a microscopic bright field image of a low-doped sample. Notice that it is not possible to distinguish micropipes from dislocations in this image since all defect features appear to be dark. Fig. 2b is a darkfield image of the same area of the wafer. Note that the two dark hexagonal objects in the center of the frame are hollow core dislocations (micropipes). In darkfield microscopy, features that reflect light in oblique angles (closed core dislocations) appear bright, and hollow core dislocations appear dark. This makes it possible to count micropipes and dislocations in undoped and SI samples.

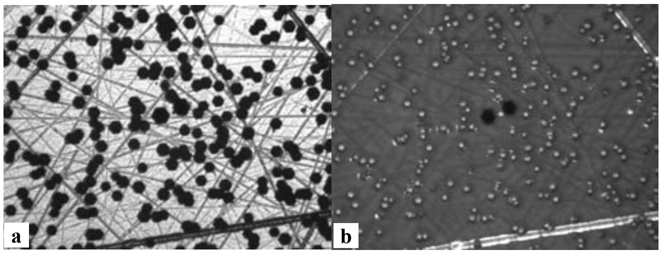

Fig. 2. Bright field (a) and dark field (b) microscopic images of an area of a low-doped 6H-SiC wafer.

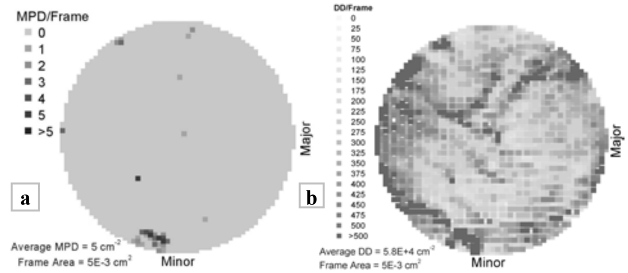

Fig. 3. (a) Micropipe density distribution map and (b) Dislocation density distribution map of a heavily doped 4H-SiC wafer (MPD = 5cm^{-2}, DD = 5.8E4 cm^{-2}).

DEFECT MAPPING

Mapping defects in SiC, especially dislocations, is important for analyzing their effects on manufactured devices. A program was written to automatically scan SiC wafers of a user-defined diameter. The operator decides the threshold value to use based on the pixel intensity of the background wafer and the defect features. This varies from wafer to wafer. Etching and subsequent polishing of wafers avoids the need for complex filtering of images, as only micropipes are present, and smaller etch pits are removed [6]. This enables the system to process image frames quickly. An image frame's area is $\approx$ 800 x 600 μm^2 for a 100X magnification. As many as 3600 frames are analyzed for 2 inch wafers with a typical exclusion zone of 2mm. It takes approximately 1.5 seconds to probe a frame area and move to the next frame area. A 2-inch wafer scan is completed in less than 2 hrs. To minimize computational storage space the frame images are automatically deleted once analyzed. An automated stage is attached to the microscope, and the coordinates of each frame are saved, along with the number of micropipes and dislocations per frame. This enables post-mapping qualification of the results, as one can return to coordinates of interest to verify the micropipe counts. This capability is very useful since micropipes and dislocations tend to cluster [4]. Since these defects form clusters and do not follow a Poisson distribution, a sampling plan cannot be used effectively for defect density estimation.

Fig. 3a is a map of a recent n^+ 2 inch 4H-SiC wafer with an MPD of 5 cm^{-2}. Fig. 3b shows a dislocation density (DD) map of the same wafer. Notice the distinct features shown in the DD map; there are clusters of dislocations around the edges of the wafer that are related to growth non-uniformities. Fig. 3a has a small region of relatively higher MPD close to the minor flat. The same region in Fig. 3b has a higher DD, demonstrating the relationship between micropipes and dislocations. Currently, all SiC production crystals grown at II-VI Inc. have a sample taken in order to map for defects and the average MPD of production grade SiC wafers is 12 cm^{-2}.

RESULTS AND DISCUSSION

The features tagged as micropipes in heavily doped SiC material are unquestionably hollow core dislocations. Additionally, when the microscope is defocused, the round features disappear abruptly whilst the hexagonal features can be trailed to the opposite face of the crystal. However, in highly compensated and undoped material, discrimination is not as simple because screw dislocations and micropipes are hexagonal, and can be similar in size. Again, when the microscope is defocused from the front-side (surface) to the backside of the wafer, the micropipe already identified under darkfield mode can be trailed through the crystal, whilst other hexagonal features (dislocations) abruptly disappear. Fig. 4 shows a set of images of an area of a wafer under darkfield mode as the microscope is defocused through the crystal, illustrating that this mode does distinguish micopipes from dislocations in etched, undoped and SI SiC wafers (Fig. 4).

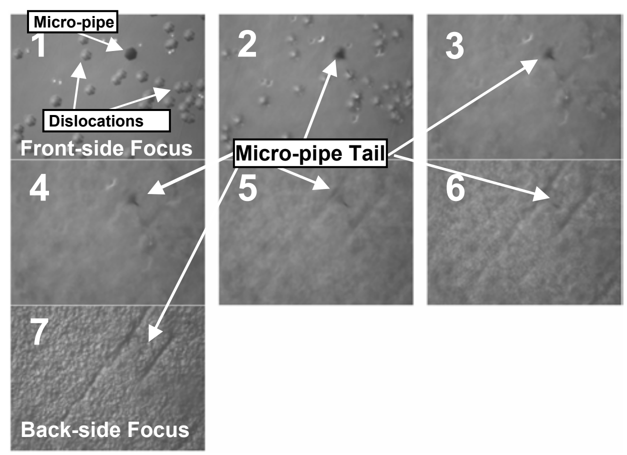

Fig. 4. Dark field images of a semi-insulating 6H-SiC as the microscope is defocused from the wafer's front-side (surface) **(1),** following the micropipe's tail **(1, 2, 3, 4, 5, 6)** until the back-side of the wafer is visible **(7)**.

To test the system's accuracy, two 6H-SiC sample wafers were evaluated with the SWBXT technique at State University of New York at Stony Brook [5]. The topography images were taken in back reflection geometry. Topography images of two regions of both samples were made. These sections were approximately 1.5 X 1.5 cm^2 in area and the topography images revealed MPDs of 45 cm^{-2} and 80 cm^{-2} for the regions evaluated. The same wafer was etched and mapped in darkfield mode and the approximate MPD for the same regions were of 41 cm^{-2} and 75 cm^{-2} respectively with the above described optical microscope system. The average MPD of the sample was 47 cm^{-2}. Fig. 5a shows the SWBXT topography image of the wafer, and Fig. 5b shows the automated MPD map of the same wafer. The second sample wafer was measured with the SWBXT to have a MPD of approximately 50 – 100 cm^{-2}, whilst the whole wafer was measured with the above described automated system to have a MPD of approximately 80 cm^{-2}. These results confirm that there is close correlation between the two techniques.

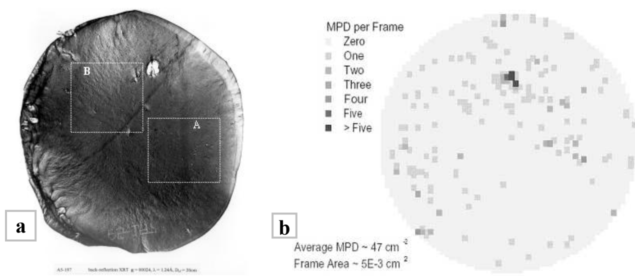

Fig. 5. SWBXT image (a) and MPD map (b) of a semi-insulating 6H-SiC wafer with similar micropipe density results. Mean MPD = 47 cm^{-2}.

The repeatability of the system was tested by allowing two users to set measurement parameters such as the threshold value, for different measurements on the same sample. The sample was prepared with the standard etch-polish regime. For clarity in the results, both operators used the same exclusion zone settings. Variation of MPD count results from these tests were < 10%. Fig. 6 shows an example of MPD maps of the same wafer tested by different users. Notice the similarity in the average MPD as well as the spatial distribution of micropipes across the wafer.

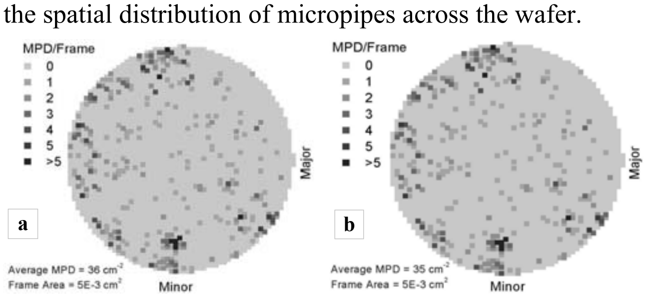

Fig. 6. MPD maps by User1 - 36cm^{-2} (a) and User2 - 35cm^{-2} (b) of a semi-insulating 6H-SiC wafer. MPD results are identical.

CONCLUSION

Accurate and automated micropipe and dislocation counting and mapping of SiC wafers has been achieved using etching in molten KOH and optical microcopy. It is a relatively simple and economical method of delineating defects.

The defects can also be characterized using other techniques including AFM, SWBXT, and optical microscopy. The SWBXT gives very accurate macro and micro images of the SiC wafer topology and defect densities. However this technique is expensive and not readily available. The resolution of optical transmission based techniques is ultimately limited to the wavelength of visible light used [4]. They can be used to count only macroscopic defects like large micropipes, but are incapable of counting defects like dislocations and smaller micropipes. In our system, these features are magnified by KOH etching ensuring the measurement of the smallest possible microspipes as well as dislocations.

The automated micropipe and dislocation counting system we have described here can map a 2-inch wafer in < 2hours with minimal computational cost. The system has been shown to be effective for wafers of different polytypes, doping concentrations and orientations. The results have been compared to those obtained by the SWBXT method, with correlation in results within < 10%. The system has also been tested to be repeatable within < 10% from run to run. The automated setup has proven to be a useful tool for manufacturing as it is currently utilized for analysis of all SiC crystals grown at II-VI Inc.

The ability to generate defect maps of SiC wafers will help to reveal the conditions under which micropipes are formed, and is helping to develop methods for their reduction and will help understand the role dislocations play in the generation and distribution of micropipes. It is hoped that cross correlation of defect maps to device performance will improve device yield.

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