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# Characterization of Dislocations in 4H-SiC

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**Keywords:** Silicon Carbide; Threading Screw Dislocation; Basal Plane Dislocation; Non-destructive Characterization; KOH Etching; X-ray Topography

**Abstract.** Accurate characterization of dislocations is crucial for optimizing the performance of SiC-based power devices. The traditional way to measure dislocation density in SiC industry is KOH etching, a destructive approach that makes the wafer no longer available for epitaxial growth. Another major limitation of this technique is the accuracy of the data since some dislocations can be hardly recognized. For example, the etch pit of threading screw dislocation is similar to that of threading edge dislocation, both of which are usually in hexagonal shape while the primary difference is the size. However, those challenges and limitations in KOH etching do not exist in X-ray topography. In this paper, the non-destructive approach, X-ray topography, is introduced to characterize dislocations in 4H-SiC industry. Threading screw dislocations were measured by both KOH etching and X-ray topography, the result of which indicates that some threading screw dislocations clearly visible in X-ray topograph are not recognizable in KOH etching image. In addition, some 60° prismatic dislocations not recognized in KOH etching image can be observed in X-ray topographs. Moreover, unlike destructive KOH etching, wafers measured by X-ray topography can be further used for annealing, epitaxial growth, ion implantation and etc., which is beneficial to SiC fundamental research.

## Introduction

4H-SiC has been widely used in power devices because of its superior physical properties compared to silicon [1, 2]. In recent years, substrate suppliers have made a lot of progress in preparing wafers with large diameter and low dislocation density [3, 4]. Nevertheless, defects still cannot be completely removed even using the latest growth techniques. These defects such as dislocations can deleteriously influence the performance of devices and even cause failures [5, 6]. Therefore, an accurate way to characterize dislocations is in demand.

The traditional way to measure dislocation density in SiC industry is using KOH etching [7], where the dislocations intersecting wafer surface are revealed as etch pits. Based on the size and shape of the etch pits, threading edge dislocations (TEDs), threading screw dislocations (TSDs) and basal plane dislocations (BPDs) are recognized. However, there are some limitations of this method. Because it is destructive, the etched wafers are no longer available for epitaxial growth, resulting in additional waste of materials, which makes it more difficult to reduce the price of wafers. More importantly, recognizing TSDs and BPDs by KOH etching is not very accurate. For example, it is usually difficult to distinguish between TSD and TED despite the etch pits of TSD is bigger than that of TED, causing underestimation of TSD density [8]. When the angle between basal plane dislocation line and sample surface is small (i.e. the line direction of BPD is close to [1-100]), the etch pit of BPD dramatically changes, making it hard to recognize it. Inaccurate TSD and BPD density measurements brings more difficulty to find correlations between dislocation and die yield or device performance.

In this paper, we introduce X-ray topography (XRT) to characterize TSDs and BPDs in 4H-SiC industry. Compared to KOH etching, XRT is non-destructive, wafers after measurements can be further used. The comparison between KOH etching and XRT is shown in Table 1. The fundamentals and functions of the equipment has been previously reported [9-11]. A. Soukhojak et. al. has mentioned to use XRT in SiC industry, but the scanning speed of XRT is low [12]. With recent release from Fraunhofer IISB, the scanning time for BPD measurement is reduced to 5 minutes, making it possible to apply XRT to mass production.

**Table 1** Comparison between KOH Etching and XRT

| | KOH Etching | XRT |
|---|---|---|
| Wafer reusability | No | Yes |
| TSD accuracy | Low | High |
| BPD accuracy | High | High |
| Workflows | Complicated | Simple |
| | 1.Heating | 1.Image Recording |
| | 2.Etching | 2.Dislocation recognition |
| | 3.Wafer Cleaning | |
| | 4.Image Recording | |
| | 5.Dislocation Recognition | |

## Experiment

The samples used in this study are commercially available 6-inch and 8-inch N-type 4H-SiC wafers with 4° off-cut towards 11-20 grown by physical vapor transport (PVT) method. The typical growth temperature is around 2300-2400°C. The Nitrogen doping concentration of the wafer is in the mid-$10^{18}cm^{-3}$ range. KOH etching method and X-ray topography were used to characterize dislocations in the samples. KOH with $Na_2O_2$ additives were heated to a temperature around 500°C, wafers placed in a Nickel bracket were then etched for 10 minutes in molten KOH [7]. After KOH etching, optical microscope was used to record images of the etch pits on the surface of the wafer. Rigaku XRTmicron was used to take X-ray topographic images. 0008 reflection topograph and 11-20 transmission topograph were recorded using Cu $K_{\alpha 1}$ and Mo $K_{\alpha 1}$ radiation respectively. High resolution topographic images were recorded by a charge-coupled device (CCD) with a resolution of 5.4 μm/pixel. The applied scanning speed is 40mm/min. 11-20 topographs used for BPD density measurement were recorded by Hypix 3000, where the scanning time is about 5 minutes for 6-inch wafers. Software 'Topography' developed by Rigaku was used to measure TSD density, while BPD density was estimated by XRT toolbox.

## Results and Discussion

**TSD Measurement.** As mentioned before, TSD density measured by KOH etching can be less accurate since the etch pits of TSD can be similar to that of TED. Fig. 1 shows the image of dislocations in the same region revealed by KOH etching (Fig. 1 (a)) and 0008 X-ray topograph (Fig. 1 (b)). TSDs or threading mixed dislocations (TMDs) are shown as dark dots in X-ray 0008 topograph (Fig. 1 (b)), as marked by the boxes. However, only the ones marked by yellow boxes show relatively big hexagonal etch pits in Fig. 1 (a), which distinguishes TSDs from TEDs. The other ones marked by red boxes do not show obviously big etch pits, making them mixed together with TEDs, thus causing it difficult to distinguish them from TEDs. Therefore, the TSD density measured by KOH etching is usually underestimated.

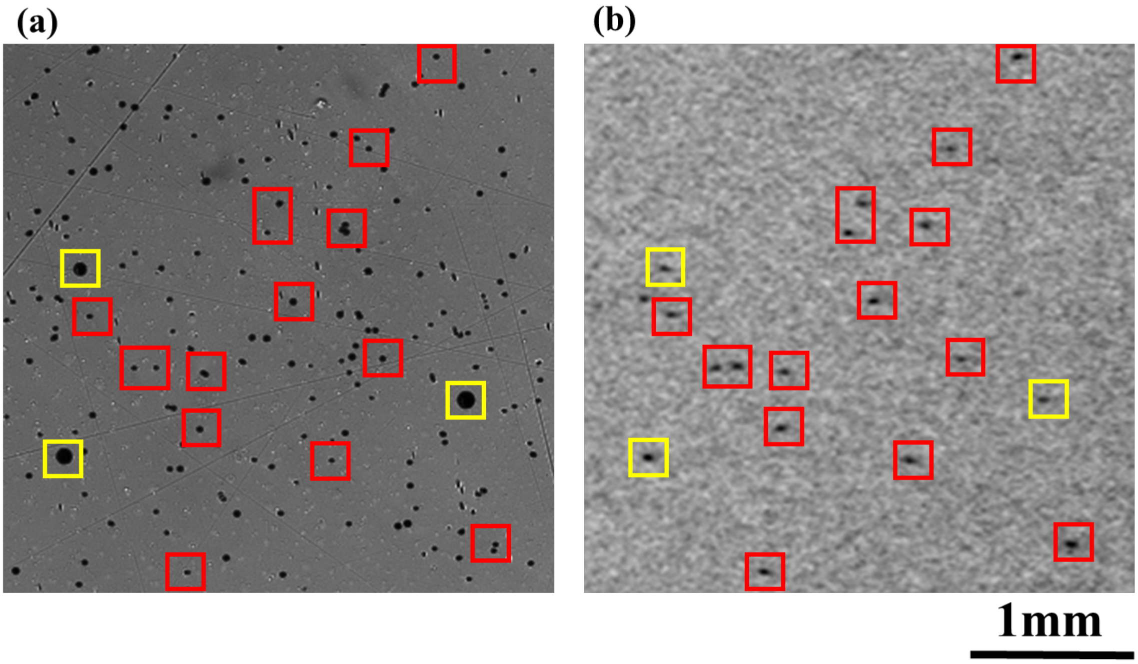


**Fig. 1.** KOH etching image (a) and 0008 X-ray topograph (b) of the same region on the same 4H-SiC wafer Si-face. Dislocations marked by yellow boxes in X-ray topograph can be recognized as TSDs in KOH etching image, while the ones marked by red boxes in X-ray topograph can be hardly recognized as TSDs in KOH etching image since they are similar to nearby TEDs.

In addition, KOH etching results usually vary from etching conditions or even crystal growth conditions. It has been reported that the etch pits size of dislocations increases linearly with the increase of mass ratio of $Na_2O_2$ additives [13]. Other effects such as etching time, wafer doping concentration or doping type are also reported to influence the shape and size of the etch pits [14]. In this study, different conditions were applied to two sister wafers. Prior to KOH etching, wafer in Fig. 2(a) was pre-processed at high temperature. The TSD revelation of these two wafers after KOH etching turns out to be quite different. As shown in Fig. 2, low angle grain boundaries on the left side of the image can be regarded as a reference point. TSDs marked by yellow boxes are revealed in both wafers as the etch pits are obviously bigger than those of TEDs. However, TSDs marked in red boxes in Fig. 2 (a) are not recognizable in Fig. 2 (b). Similarly, some TSDs in Fig. 2 (b) are not recognizable in Fig. 2 (a) either. This result indicates that TSD density and distributions measured by different KOH etching recipes can be significantly different, a more accurate and consistent method is in demand.

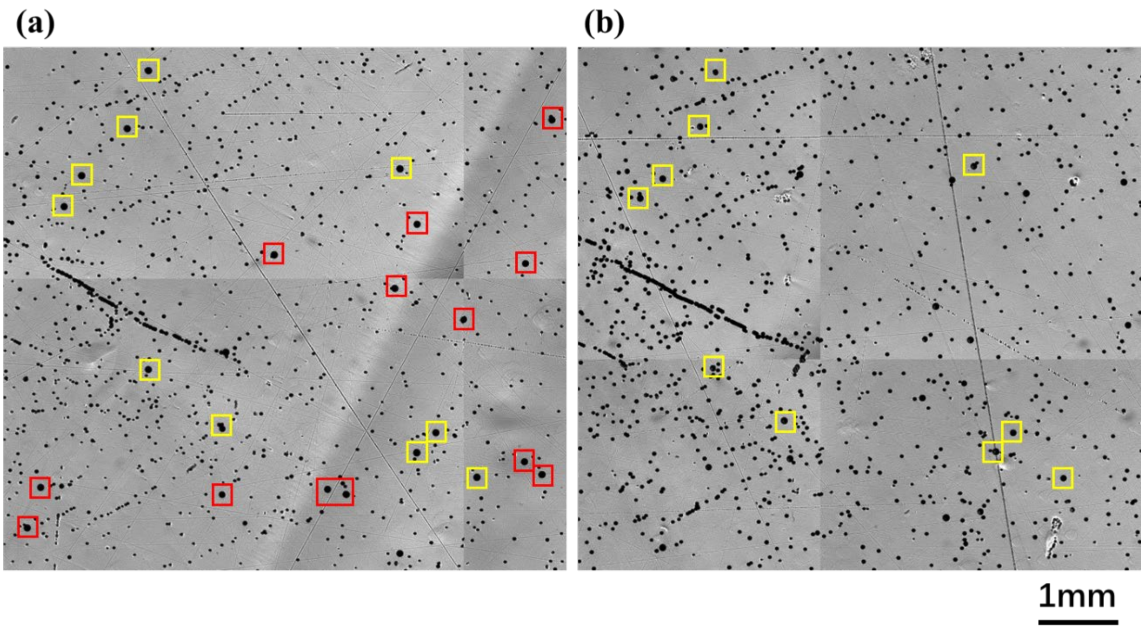


**Fig. 2.** KOH etching image of two sister wafers right next to each other ((a) & (b)). Two different etching conditions were applied to these two wafers. Etch pits marked by yellow boxes are TSDs revealed in both wafers, while the etch pits marked by red boxes are bigger only in left image.

Compared to KOH etching, X-ray topography is a more stable and consistent approach to characterize TSDs. Since $g \cdot b = 0$ and $g \cdot b \times l = 0$, where $g$ is the normal of the diffraction plane, $b$ is the Burgers vector of the dislocation, and $l$ is dislocation line direction, TEDs (the Burgers vector is 1/3<11-20> while line direction is along c axis) is out of contrast in X-ray 0008 reflection, identification of TSDs is thus not to be confused with TEDs in XRT [15, 16]. The consistency of XRT is evaluated by measuring sister wafers from the same boule. Since TSDs are growth dislocations that are inherited from the seed crystal and grow along 0001 direction, their density should not vary significantly from the first wafer on the seed side to the last wafer on the dome side. Although some TSDs can be converted to Frank dislocations during growth [17], the converted ones are usually a small fraction of all the TSDs, so the TSD density of the wafers from the same boule should be almost the same. Fig. 3 shows the TSD density of the wafers from the same boule measured by XRT. In order to eliminate the error caused by chance, three boules were measured. The measured TSD density does not change significantly for the same boule, indicating the stability and consistency of the data recorded by XRT. Moreover, the repeatability and reproducibility of XRT are evaluated by measuring the same wafer for multiple times. As shown in Fig. 4, TSD density almost stays the same under different measurements (the error is less than 5%), indicating good stability and repeatability of this characterization method.

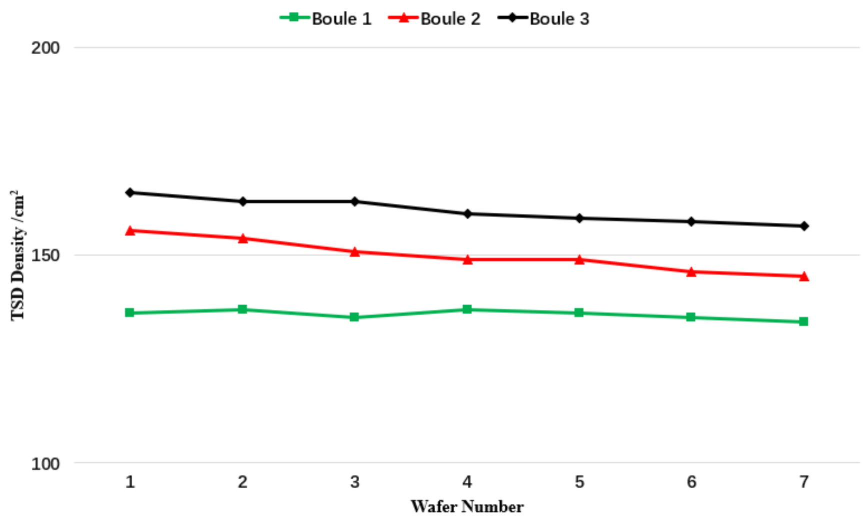


**Fig. 3.** TSD density of the wafers from the same boule measured by XRT. Wafer 1 to 7 is from the same boule, where wafer 1 is close to the seed side while wafer 7 is close to the dome side.

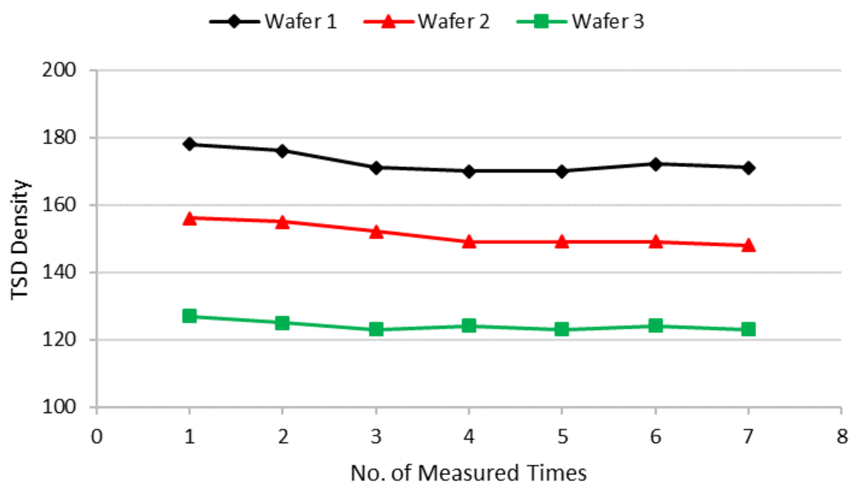


**Fig. 4.** Evaluation of XRT repeatability and reproducibility. TSD density of three wafers measured by XRT for 7 times.

**BPD measurement.** In 4° offcut 4H-SiC, BPDs are usually revealed as shell-shape etch pits in KOH etching. However, that is not always the case. When BPD is almost parallel to 11-20 direction (BPD 1 in Fig. 5 (a)), the angle between dislocation line and wafer surface is about 4°, and the etch pit of BPD can be easily recognized. But if BPD is close to 1-100 direction (BPD 2 in Fig. 5 (a)), this angle is close to zero, resulting in unrecognizable etch pit. In real case, the line direction of BPD is random, once the angle between BPD line direction and wafer surface is reduced to 1 or 2°, the recognition of BPD etch pits turns to be difficult as their shape changes dramatically, resulting in underestimation of BPD density. As shown in Fig. 5 (b), BPDs marked by red boxes are recognizable, where the etch pit is like a shell. However, the BPDs marked by yellow arrows are not recognizable since the etch pit is like a needle, quite different from other standard BPD etch pits. Those ones are most likely 60° prismatic dislocations based on their line directions and distributions (60° is the angle between dislocation line direction and [11-20]) [18].

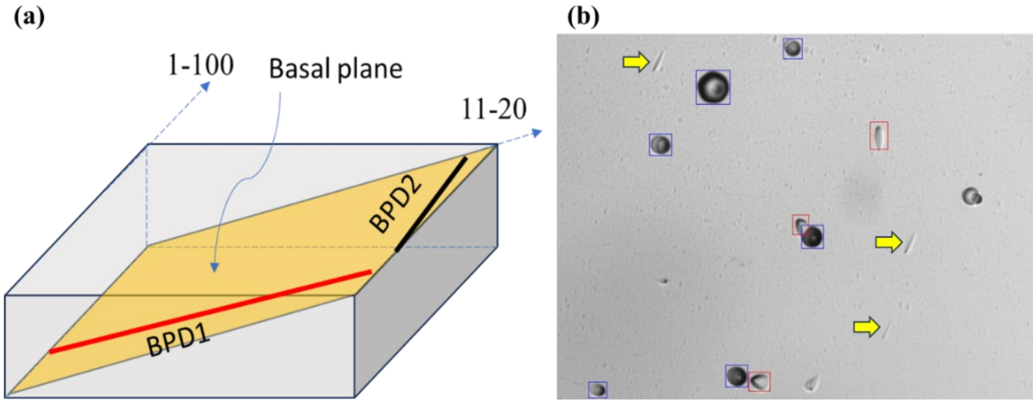


**Fig. 5.** (a) A diagram showing BPDs with different line directions. (b) KOH etching image of 4H-SiC, where BPDs are marked by red boxes, while the yellow arrow marked BPDs are not recognized.

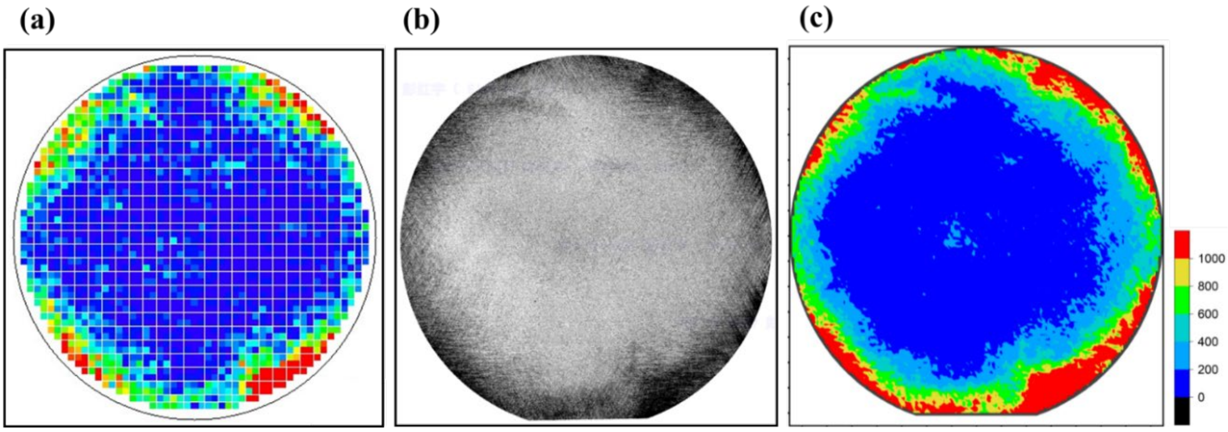


**Fig. 6**. KOH etching map (a), 11-20 X-ray topograph (b) and corresponding density map (c) of BPDs of the same wafer.

Since some of the BPDs (specifically 60° prismatic dislocations) can be hardly recognized in KOH etching, the dislocation density can be underestimated and the distributions can be inaccurate. Fig. 6 (a) is a KOH etching map of BPDs in a typical 6-inch wafer, where the density is very low at the position of 3, 6, 9 and 12 o'clock, and the overall BPD density is 170/cm$^2$. However, as shown in 11-20 X-ray topograph (Fig. 6 (b)), there are many 60° prismatic dislocations in those four regions. Fig. 6 (c) is the BPD density map estimated from XRT, where the density in those four regions is higher compared to that estimated from KOH etching and the overall BPD density is 303/cm$^2$. The omission of some 60° prismatic dislocations in KOH etching leads to the underestimation of BPD density, and that is also one of the reasons why a specific BPD distribution (higher BPD density at 4 corners) is usually obtained in KOH etching map.

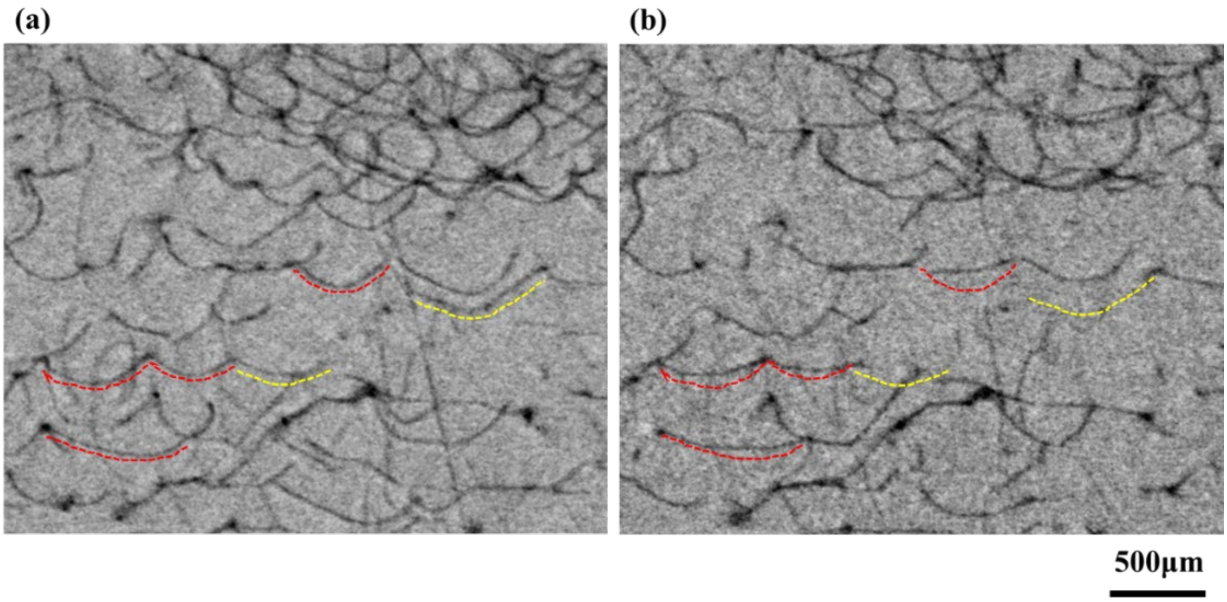


**Fig. 7.** 11-20 X-ray topograph of the same wafer before (a) and after (b) annealing.

**Non-destructive.** In addition to being able to measure dislocation density with high accuracy, XRT is also non-destructive, which not only saves wafers, but also has advantages in many other aspects. For example, the wafer can be imaged before some processes such as annealing, epitaxial growth and ion implantation. After those processes, the same wafer can be imaged again by XRT, therefore, dislocation behaviors during these processes can be investigated. Fig. 7 (a) & (b) are 11-20 X-ray topographs of 4H-SiC wafer before and after annealing. As marked by the dashed red curves, some BPDs get straighten after annealing to minimize line tension, indicating that some energies stored in the wafer could be released during annealing process. Some BPDs marked by dashed yellow curves moves during annealing and could not be observed in its original position after the process.

## Summary

Characterization of TSD and BPD has been discussed in this paper. The density of both TSD and BPD can be underestimated in traditional KOH etching since some etch pits of TSDs are similar to that of TEDs, and some etch pits of 60° prismatic dislocations can be hardly recognized. Moreover, KOH etching results are sensitive to etching conditions, same wafer could show different dislocation etch pits under different etching recipes. Since KOH etching is destructive, etched wafers are no longer available for epitaxial growth. But these limitations in KOH etching do not exist in X-ray topography. TSD is not to be confused with TED in XRT because of $g \cdot b = 0$ criteria and prismatic dislocations can be simply revealed by XRT. And XRT has good repeatability and reproducibility. As XRT is non-destructive, the measured wafers can be further used for annealing, epitaxial growth, ion implantation and etc. Therefore, we propose to apply XRT in SiC industry to replace traditional KOH etching, from which wafers can be saved, helping to reduce substrate cost. More importantly, accurate dislocation data generated by XRT helps to correlate substrate defects with epitaxial defects and device performance, providing important information for figuring out root causes of epitaxial defects or device failures.

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