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Characterization of Prismatic Slip in SiC Crystals by Chemical Etching Method

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Abstract. In 4H-silicon carbide crystals, basal plane slip is the predominant deformation mechanism. However, prismatic slip is often observed in single crystals grown by the physical vapor transport method as the diameter expands to 6 inches or larger. Thermal modeling has shown that occurrence of prismatic slip is attributed to increased radial thermal gradients. While X-ray topography can be used to characterize the presence and extent of prismatic slip, the feasibility of using the chemical etching method to assess the extent of prismatic slip in an industrial setting is investigated. The distribution of scallop shaped etch pits oriented along the $\langle 11\bar{2}0\rangle$ directions that correspond to prismatic dislocations, correlate well with the results of the thermal model that predicts the occurrence of prismatic slip dislocations. This capability of the etch pit method to characterize prismatic slip can be used to manage radial thermal gradients during PVT growth.

Introduction

Due to its excellent electrical and thermal properties, wide band gap semiconductor silicon carbide (SiC) is replacing conventional silicon materials for high-temperature, high-voltage, and high-frequency applications. However, the quality of the bulk SiC materials has been an issue to prevent the wide application of SiC-based power devices as the crystalline defects can affect the reliability of such devices [1]. Considerable progress has been made to reduce the defect densities, while wafers up to 8 inches in diameter are being developed.

As the crystals become larger in size, one big challenge is the control of radial thermal gradients in the physical vapor transport (PVT) growth chamber. Failure to achieve optimal thermal fields can activate prismatic slip systems, which are $\{1\bar{1}00\}\langle 11\bar{2}0\rangle$ in 4H-SiC crystals, even though prismatic slip systems are often harder to be activated than basal plane slip systems ($(0001)\langle 11\bar{2}0\rangle$). The screw oriented prismatic dislocations can subsequently cross slip onto the basal planes. Despite the fact that the majority of basal plane dislocations (BPDs) will be converted to less harmful threading edge dislocations (TEDs) during the subsequent epitaxial growth, it is still important to avoid the activation of prismatic slip systems. It has been reported that screw type BPDs, especially with Burgers vectors along the offcut direction, can multiply and replicate into epilayers during homoepitaxy instead of being converted to TEDs, causing degradation of device performance [2]. Observation of prismatic slip of threading edge dislocations (TEDs) (Fig. 1) have been made in either axially cut 4H-SiC boules [3] or c-plane 4H-SiC wafers with 4-degree offcut angle using synchrotron white-beam X-ray topography (SWBXT) [4]. Moreover, a model has been developed to predict the non-uniform distribution of 3 different prismatic slip systems across all the regions in the crystal and provide estimation of radial thermal gradient-induced thermal stresses during the bulk crystal growth process.

This model has been applied successfully in both 4H-SiC and 2H aluminum nitride crystals grown by the PVT method [4, 5].

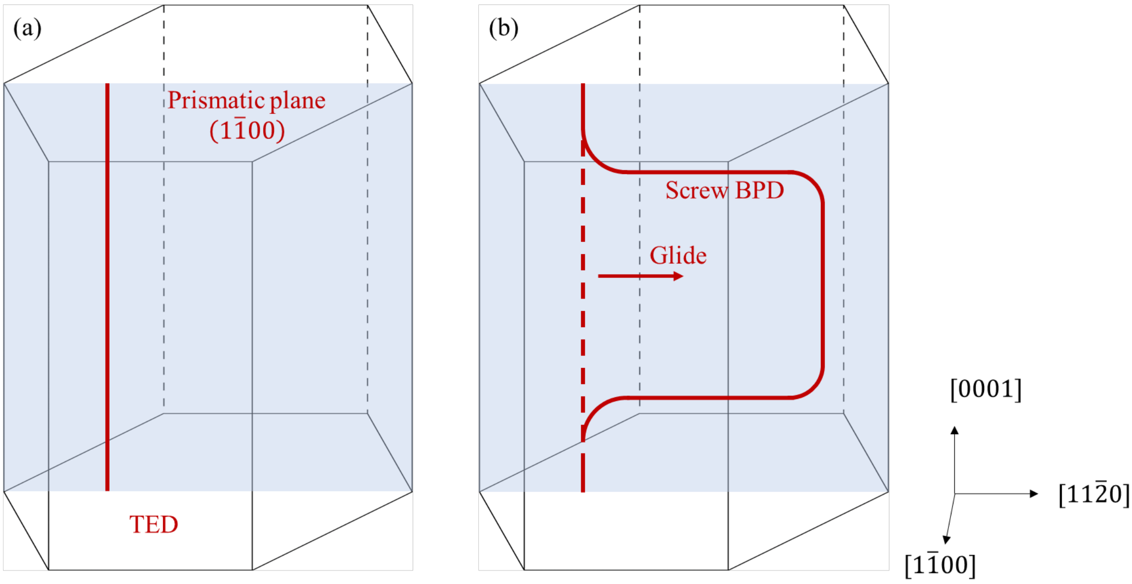

Fig. 1. Schematics demonstrating prismatic slip in 4H-SiC. (a) TED lying in the prismatic plane; (b) Glide of the TED in the prismatic plane leaving behind screw dislocation segments that can cross slip on to the basal plane.

Despite its ability to directly observe prismatic slip dislocations, X-ray topography characterization can be limited in its availability for use in industry. Therefore, it is important to employ readily carried out dislocation characterization techniques such as the wet etching method to help assess the extent of prismatic slip in PVT grown SiC crystals and optimize the temperature gradients. Molten KOH etching [6] is the most popular wet chemical etching method due to its high efficiency and convenience. Dislocations that lie in the basal plane and intersect with the surface will be shown as scallop shaped pits after etching (Fig. 2). It is also noted that the orientation of the etch pits can be used to determine the line direction of the BPD. This correlation allows the possibility of using molten KOH etching to characterize the prismatic slip systems in PVT grown 4H-SiC crystals. In this work, correlation between the orientations of scallop shaped pits from molten KOH etching and the distribution of prismatic dislocations revealed by SWBXT is analyzed and the ability to use wet etching method to characterize prismatic slip dislocations in PVT grown SiC crystals is evaluated.

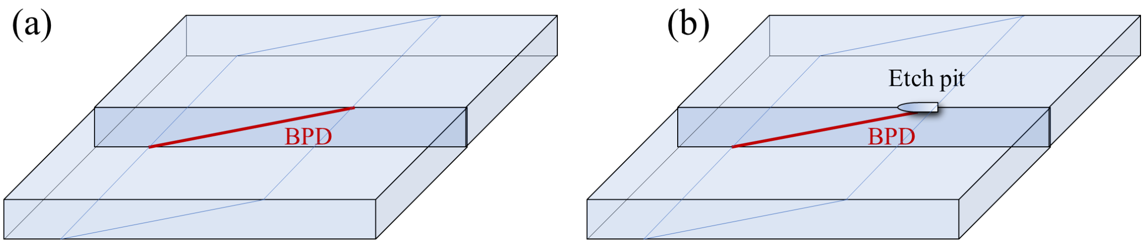

Fig. 2. Schematics showing (a) Intersection of a screw type BPD (or screw type prismatic dislocation) with the surface of the 4° off-cut 4H-SiC wafer; (b) Longer axis of scallop shaped etch pit produced by KOH etching at the site of emergence of BPD is parallel to the line direction of BPD.

Experimental Procedures

Two commercially available 150-mm 4H-SiC wafers produced using the same growth conditions (samples A and B) were selected for this study. Sample A was characterized by SWBXT and sample B was characterized by wet etching method. The SWBXT experiments were carried out in transmission geometry, as shown in Fig. 3. The 11$\bar{2}$0 diffraction spot is selected as all types of prismatic dislocations are visible, and a topographic image was recorded by scanning the sample with the white X-ray beam. Agfa Structurix D3-SC high-resolution X-ray films were used to collect the diffraction data.

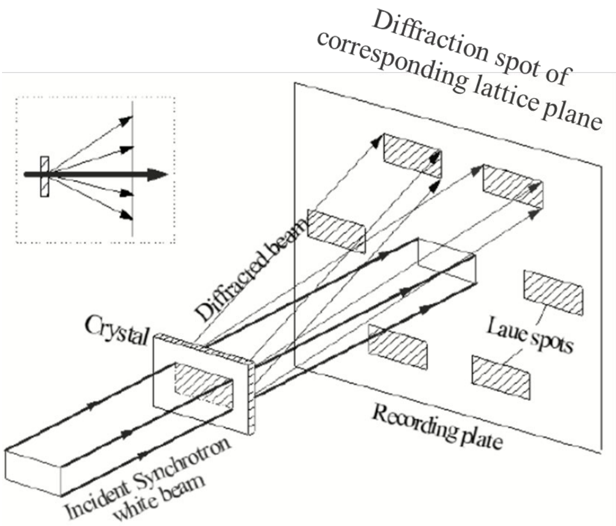

Fig. 3. Schematic of SWBXT in the transmission geometry.

Wet chemical etching was conducted on sample B, using molten KOH. The wafer was etched at 520 °C for 10 minutes.

Results and Discussion

Fig. 4 shows the overall $11\bar{2}0$ transmission topograph of sample A. Prismatic slip can be observed around the periphery region as marked in the figure. In the 12 magnified images shown in Fig. 5 (a), prismatic slip dislocations are characterized as oriented linear contrast, which is predominant among all the dislocation contrast. The bands of parallel dislocations oriented along the $<11\bar{2}0>$ directions are the screw oriented prismatic dislocations. As they maintain their linear configurations, this indicates that cross slip of the prismatic slip dislocations has not taken place. 2 of 3 different prismatic slip systems are observed in each region, which can be correlated to the predictions from the model shown in Fig. 5 (b). As thermal stresses induced by radial temperature gradients during crystal growth is the main cause of prismatic slip in 4H-SiC, the existence of prismatic slip dislocations indicates that the radial thermal profile in the growth chamber needs to be optimized.

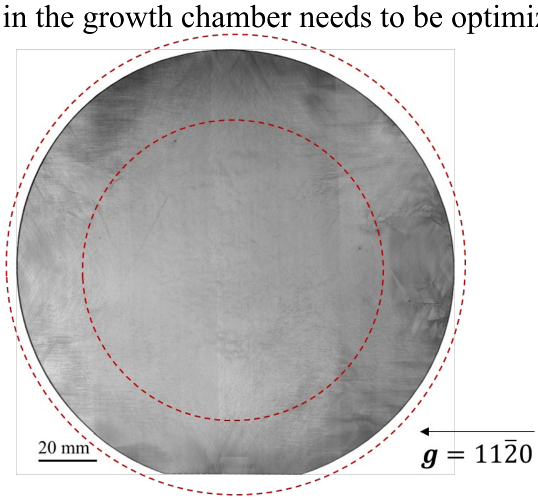

Fig. 4. Transmission X-ray topograph of the 6” wafer of sample A. The concentric red-dotted circles enclose the regions characterized by prismatic slip.

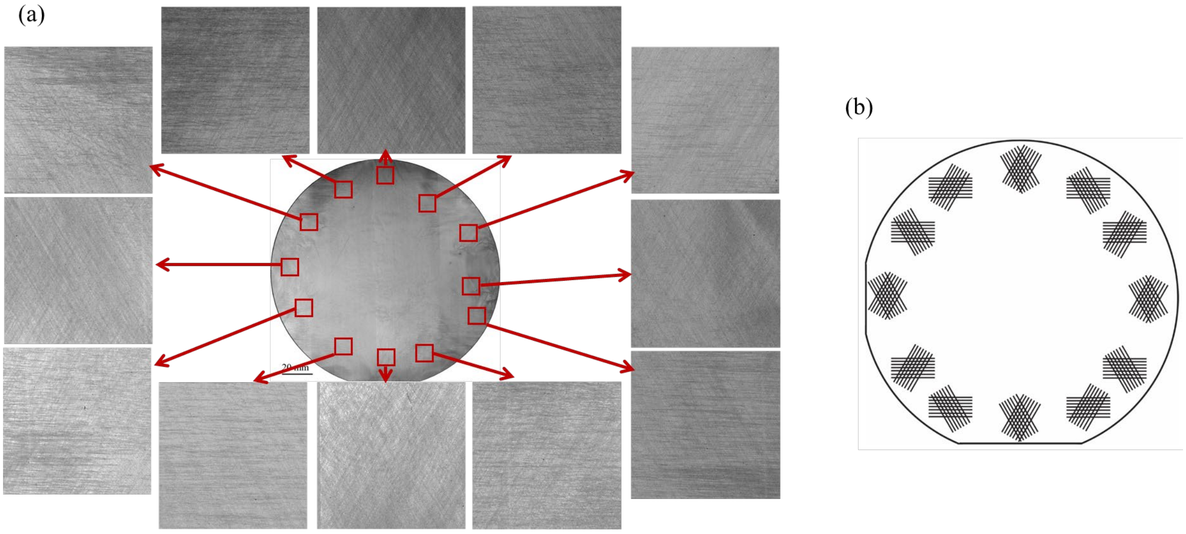

Fig. 5. (a) Magnified images from 12 different periphery regions on sample A. (b) Prediction of the prismatic slip dislocations configuration in the 12 different regions. The prismatic slip directions on the topographs in each region match the predictions in for that region.

The micrographs of 12 regions on sample B, which was etched by KOH under 520 ℃ for 10 minutes, were selected corresponding to the 12 regions in Fig. 5 (a). As the wafer has a 4-degree offcut towards $11\bar{2}0$ direction, dislocations on the basal plane, i.e., (0001) plane, intersect with the surface of the sample to the right. Therefore, the tips of the scallop shaped etch pits from prismatic slip dislocations are predicted to have 3 possible directions ($[\bar{1}2\bar{1}0]$, $[11\bar{2}0]$ and $[2\bar{1}\bar{1}0]$), which is represented by the arrows in Fig. 6 (a). Fig. 6 (b) is a selected image from region 1 of sample B after etching. The majority of scallop shaped etch pits fall in one of the possible orientations predicted by Fig. 6 (a), as marked by the arrows. The prismatic slip systems observed in this region are $(01\bar{1}0)[2\bar{1}\bar{1}0]$ and $(10\bar{1}0)[\bar{1}2\bar{1}0]$. This observation agrees with the fact that prismatic dislocations are present in sample A, which is from the same growth conditions as sample B. Fig. 6 (c) and (d) show the same analysis in region 2 and region 3 on the wafer. Region 2 shows prismatic slip systems of $(\bar{1}100)[11\bar{2}0]$ and $(01\bar{1}0)[2\bar{1}\bar{1}0]$, and region 3 shows $(\bar{1}100)[11\bar{2}0]$ and $(10\bar{1}0)[\bar{1}2\bar{1}0]$. In these regions, all 3 types of prismatic slip systems are revealed by the etch pits. Generally, the line directions of regular BPDs in 4H-SiC are random. Therefore, some of the marked etch pits might come from regular BPDs instead of prismatic slip dislocations. However, considering that most of the etch pits observed are oriented along the predicted directions, it can be concluded that prismatic slip dislocations dominate among the dislocations on the basal plane.

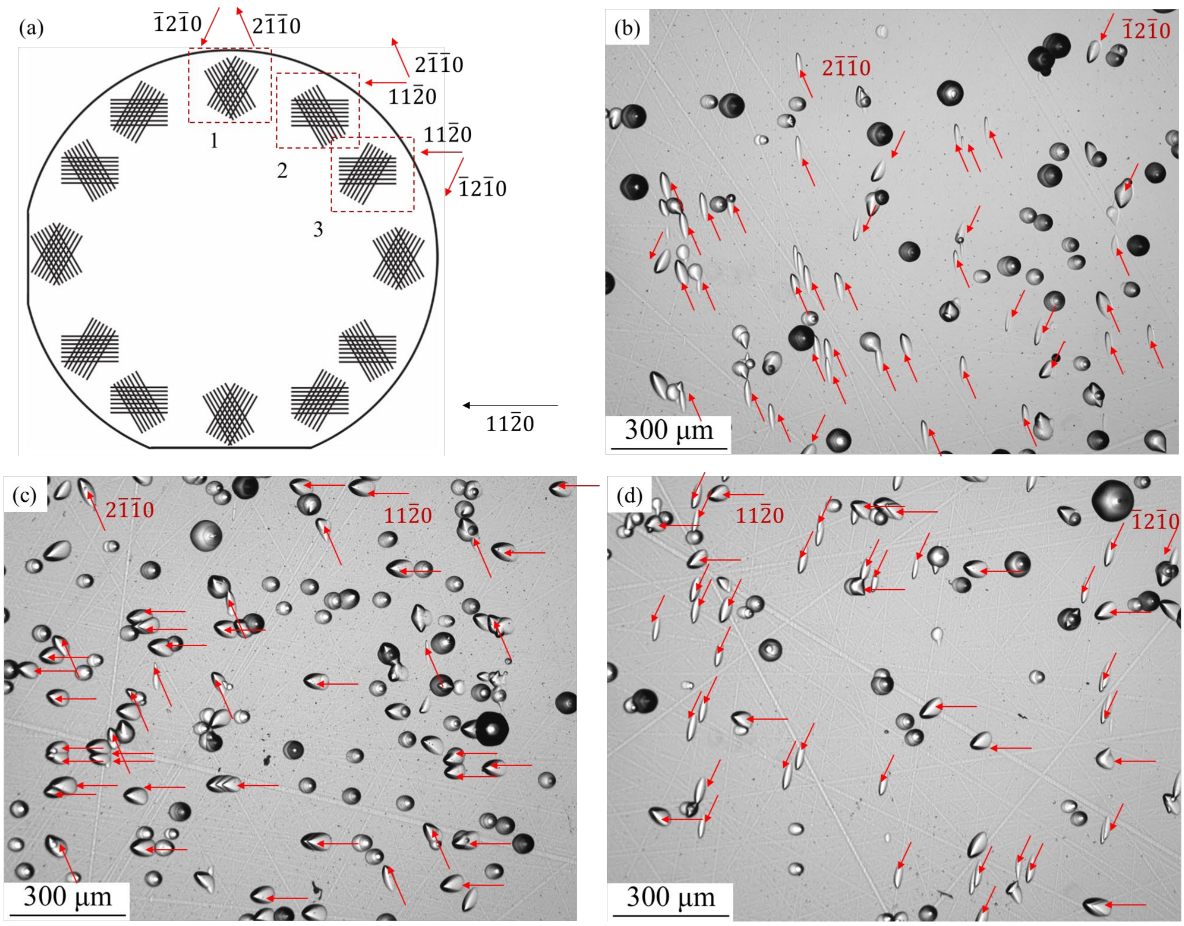

Fig. 6. (a) Positions 1-3 marked on the predicted prismatic slip configuration diagram. (b-d) Magnified images showing etch pits from position 1-3 on sample B. Orientations of etch pits in each image match the predicted prismatic dislocation orientations.

The same analysis was conducted for the other regions. Fig. 7 summarizes all 12 regions on the wafer. The configuration of etch pits on sample B fits the prediction by the model, which is marked by the arrows in the same way as in Fig. 6. This observation also matches the SWBXT topography results from sample A very well in that prismatic slip dislocation is the major contributor to BPDs. It is noted that a significant portion of the prismatic slip dislocations are with Burgers vectors along the offcut direction, which tend to replicate into the epitaxial layer, causing degradation to the devices fabricated [2]. Therefore, it is important and useful to adopt wet chemical etching in characterizing prismatic slip dislocations and optimize growth to limit their extent.

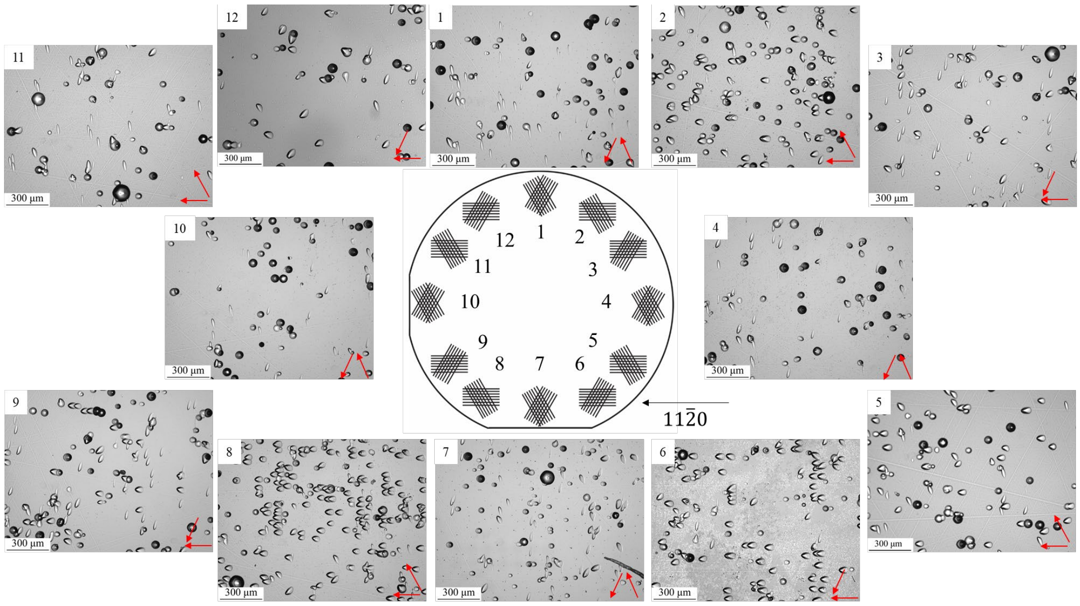

Fig. 7. Magnified etching profiles from all 12 regions on sample B.

Summary

In this paper, the capability of wet etching method by molten KOH to reveal prismatic slip dislocations in 4H-SiC crystals grown by PVT method is demonstrated. With the support of a previously developed thermal model that predicts the configuration of prismatic dislocations and SWBXT observations, it is confirmed that the highly oriented etch pits are produced by dislocations belonging to the prismatic slip systems activated during the growth. The wet etching method is an easy way to characterize prismatic dislocations when X-ray topography is not available. Therefore, it is useful to evaluate the radial temperature gradients during the growth process.

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