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Structural characterization of the growth front of physical vapor transport grown 4H-SiC crystals using X-ray topography

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ABSTRACT

The defect structure at the growth front of 4H-SiC single crystal boules grown by the physical vapor transport (PVT) growth method has been investigated using X-ray topography. Plan-view observations of the growth front of the boules revealed that basal plane dislocations (BPDs) were often nucleated at the outcrops of threading screw dislocations during growth. The observations also revealed that BPDs introduced in the facet and near-facet regions were hardly multiplied during the PVT growth process, and thus the crystal portions just beneath these regions contained very low densities of BPDs. The cross-sectional observations of the grown boules confirmed this result and further revealed that a number of BPDs were introduced in the crystal portions well distant from the growth front. They are thought to be introduced *via* dislocation multiplication due to large thermo-elastic stresses imposed on the shoulder parts of the grown boules during PVT growth.

1. Introduction

Defect formation during physical vapor transport (PVT) growth of bulk SiC crystals is still a major obstacle for realizing high performance SiC power devices. Particularly, formation of dislocations is a serious problem since certain types of dislocations are detrimental to the yield and reliability of SiC power devices. Dislocations in PVT-grown SiC crystals are broadly classified into two types. One is threading dislocations extending along the *c*-axis, and the other is basal plane dislocations (BPDs) lying in the basal plane. Threading dislocations, particularly threading screw dislocations (TSDs) degrade the blocking capabilities of SiC diodes [1], whereas BPDs have a serious impact on the reliability of SiC bipolar devices [2] as well as unipolar devices such as SiC MOSFETs and JFETs [3,4]. Therefore, significant effort has been dedicated towards the reduction of TSD and BPD densities.

A marked difference between TSDs and BPDs exists in their formation processes during PVT growth of SiC crystals. Most TSDs are inherited from the seed crystal and often formed at the initial stage of crystal growth [5]. BPDs could also be inherited from the seed crystal and formed at the initial seeding process, but they do not propagate through the entire crystal since their extension directions are limited in the basal plane, almost normal to the growth direction. Therefore, a high density of BPDs existing in the top and middle portions of SiC crystals cannot be explained by the above-mentioned mechanisms, and the causes of BPDs observed in the portions are still poorly understood.

One possible explanation is that BPDs would be nucleated at the growth front (growing surface) and then incorporated into grown crystals. In general, defect formation at the growth front is closely related to the shape and morphology of the growing crystal surface; the crystal shape determines the magnitude and distribution of the thermoelastic stress imposed on the grown crystals [6–8], and the surface morphology at the growth front largely affects the defect formation kinetics. Therefore, control of these growth parameters is crucial to obtain high-quality SiC single crystals.

In this paper, we characterized the defect structure at the growth front of 4H-SiC boules grown using the PVT growth method. This study focused to identify where and how BPDs are nucleated and multiplied at the growth front. The growth front comprises the $(000\bar{1})$ facet and its outer (non-facet) regions; they exhibit characteristic surface morphologies in terms of the step-terrace structure. The morphologies of these two regions are quite different [9,10], and thus different defect formation kinetics would govern the two surface regions. Furthermore, these two regions grow into different crystal shapes; the facet is fairly flat and has a temperature distribution along the surface during PVT growth [10], whereas the non-facet regions are convex-shaped toward the growth direction and are assumed to be roughly isothermal during growth. The degree of this convexity would also be a crucial parameter for defect formation during PVT growth of SiC. As such, investigation of

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the crystalline properties of the facet and non-facet regions at the growth front provide valuable information about the defect formation during PVT growth of SiC crystals.

2. Experimental procedure

To characterize the growth front of SiC boules, one-inch and two-inch diameter 4H-SiC single crystal boules were grown on an on-axis or off-oriented $(000\bar{1})$ 4H-SiC seed crystal using the PVT growth method. The typical growth temperature was 2300–2400 °C, and the argon gas pressure was maintained between 1.0 and 2.0 kPa during growth. The grown boules were nitrogen-doped and contained nitrogen donors in the mid-10^{18} cm^{-3} range. The surface morphology of the growth front of as-grown boules was examined by differential interference contrast (DIC) optical microscopy. The structural characterization of the growth front was conducted *via* X-ray topography. Reflection and transmission X-ray topographs were obtained using Cu Kα and Mo Kα radiations, respectively. The topographs were recorded using a charge-coupled device (CCD) camera with a pixel size of a few micrometers. A computer controlled detection system took multiple several-millimeter-sized topographic images that were stitched together to form topographic images of a few centimeter size. Reflection X-ray topographs were taken in plan-view imaging of the as-grown surface (growth front) of 4H-SiC boules with asymmetric $11\bar{2}\bar{8}$ and $1\bar{1}0\bar{7}$ reflections. In addition to these plan-view observations, cross-sectional observations were also conducted; the grown boules were sliced vertically into $(1\bar{1}00)$ wafers, which were examined by transmission X-ray topography with $11\bar{2}0$ and $000\bar{8}$ diffractions.

3. Results and discussion

3.1. Plan-view observations of defect structure at the growth front

Fig. 1 shows a DIC optical microscope image of the growth front of a one-inch diameter nitrogen-doped 4H-SiC boule examined in this study. The figure reveals that the growth front of the nitrogen-doped (mid-10^{18} cm^{-3}) boule comprises three distinct morphological regions: the $(000\bar{1})$C facet (denoted as F in the figure) and non-facet (NF) regions, and the intermediate region (I) between the two regions. The facet region showed hexagonal symmetry comprising six vicinal $(000\bar{1})$C surfaces tilted toward $\langle 1\bar{1}00 \rangle$ directions. The vicinal surfaces were separated by six crystallographically-equivalent ridges extending along the $\langle 11\bar{2}0 \rangle$ directions. The non-facet region showed macroscopically smooth morphology, and the convexity of the region varied depending on the growth conditions, particularly the temperature distribution in the growth cell. The intermediate region was arranged on the perimeter of the facet region. The region was narrow and macroscopically exhibited a slightly rough morphology.

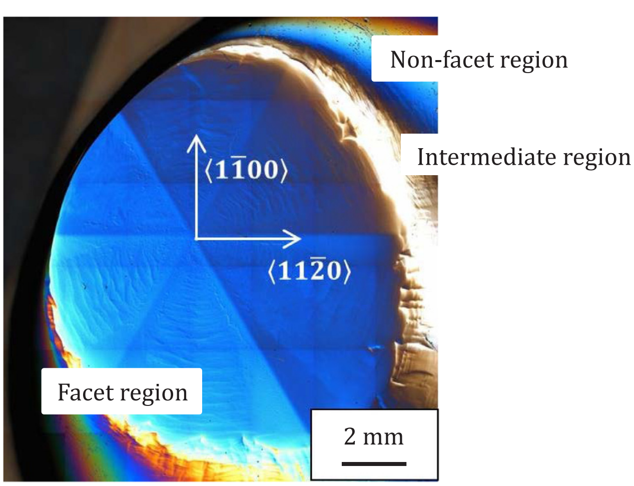

Fig. 1. DIC optical microscope image of the growth front of a one-inch diameter nitrogen-doped (mid-10^{18} cm^{-3}) 4H-SiC boule examined in this study. The growth front consists of three distinct morphological regions: the $(000\bar{1})$C facet and non-facet regions, and the intermediate region between them.

As revealed in Fig. 1, the shape of the growth front is different between the facet and non-facet regions, and thus the defect formation could be different between the two regions. To examine the defect structure across the boundary between the facet and non-facet regions, we took X-ray topographic images of the as-grown surface of 4H-SiC boules. Fig. 2 shows reflection X-ray topographs with (a) $11\bar{2}\bar{8}$ and (b) $1\bar{1}0\bar{7}$ diffractions. In the figure, the facet, non-facet, and intermediate regions are denoted as F, NF, and I; their boundaries are marked by dashed lines. Both topographs contain relatively intense linear contrasts roughly extending along the $\langle 1\bar{1}00 \rangle$ directions; some of them are marked with open triangles in the topographs. They correspond to small angle grain boundaries consisting of threading edge dislocations (TEDs) penetrating the crystal along the *c*-axis. We also observed small dot-like contrasts in both topographs. They have two possible origins, i.e., TSDs and TEDs, but we ascribed them to TSDs based on their estimated density ($\sim 10^3$ cm^{-2}). As seen in Fig. 2, no marked difference in the defect structure was found among the three regions of the as-grown surface, *i.e.*, the facet, intermediate, and non-facet regions, implying that the facetted growth front hardly affect the formation of extended defects during PVT growth of 4H-SiC boules.

The topographs with $11\bar{2}8$ and $1\bar{1}0\bar{7}$ diffraction vectors shown in Fig. 2(a) and (b) exhibit slightly different image textures. This is thought to be due to BPDs existing in the crystal portions just beneath the as-grown surface of 4H-SiC boules. BPDs, which have Burger vectors within the basal plane, are out of contrast when the diffraction vector is perpendicular to their Burgers vector, and thus the two topographs taken with different diffraction vectors exhibit slightly different contrast patterns (image textures).

Fig. 3 shows enlarged X-ray topographs for more detailed investigations of the BPD structures underneath the facet and near-facet regions. In the figure, wider area topographs [Fig. 3(a) and (c)] are also presented, in which the positions at which the enlarged X-ray topographs [Fig. 3(b) and (d)] were taken are indicted by open squares. The topographs shown in Fig. 3(a) and (b) were taken with $11\bar{2}\bar{8}$ diffraction, whereas those in Fig. 3(c) and (d) were taken with $1\bar{1}0\bar{7}$ reflection. Fig. 3(b) and (d) were acquired from the same area in the as-grown crystal surface of a 4H-SiC boule [indicated by open squares in Fig. 3(a) and (c)]. In Fig. 3(b), both TSDs and BPDs were observed; they are indicated by closed and open triangles, respectively. On the other hand, only TSDs were detected in Fig. 3(d), in which BPDs showed no distinct contrasts because the diffraction vector was set perpendicular to their Burgers vectors.

It is noteworthy in Fig. 3(b) that many of the observed BPDs seem to emanate from TSDs. We examined other areas on the growth front and found similar behaviors of BPDs emanating from the outcrops of TSDs. These results suggest that the existence of TSDs would be related to the BPD formation at the growth front, and TSDs intersecting the growing surface induce the nucleation of BPDs at the surface during PVT growth of 4H-SiC boules. The mechanism is yet to be clarified at present. However, the elastic interaction between TSDs and the growing surface would play a crucial role in this phenomenon. Wang et al. reported similar behaviors of BPDs in the presence of micropipes (super screw dislocations) [11]. They found that BPDs existing in commercially available 6H-SiC substrates connect or emanate from micropipes, comprising dislocation networks in the substrates. Similar BPD structures were also reported in 4H-SiC epitaxial layers [12]. Micropipes and TSDs extending along the *c*-axis in an infinite crystal do not have a shear stress component parallel to the basal plane, and hence they could not be the causes of BPDs in an infinite crystal. However, in a finite crystal, they elastically interact with the free surface, and the resultant surface relaxation due to the image force effect of dislocations [13] can

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