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Studies of Defects and Strains Using X-Ray Topography and Diffraction

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Summary

A short review is presented of recent Synchrotron White Beam X-ray Topography (SWBXT) and High Resolution Triple-Axis X-ray Diffraction (HRTXD) studies of defects and distortions in 4H and 6H SiC substrates, and homo- and hetero-epitaxial layers grown on these substrates. In the substrates, defects observed include closed-core and hollow-core screw dislocations (micropipes) in 6H and 4H, deformation induced basal plane dislocations in 6H and 4H, and small angle boundaries in 4H. For the hetero-epitaxial layers, consisting of 3C grown on specially prepared 4H and 6H mesas, detailed correlation between the defect content of the mesas and the choice of 3C variant and the subsequent lattice mismatch between heteroepilayer and substrate is presented.

Introduction

Over the past decade or so, Synchrotron White Beam X-ray Topography (SWBXT) [1] has played a very strong role in the development of the wide bandgap semiconductor material silicon carbide (SiC) [2,3]. In particular, it has demonstrated that the "micropipe" defect is a hollow-core screw dislocation; those with Burgers vectors of 3*c* or larger usually have hollow-cores while those with Burgers vectors less than 3*c* do not. These screw dislocations are classic "growth dislocations" in that they thread the growing crystal interface and are continued via replication as the crystal grows. When Scanning Electron Microscopy was used to measure micropipe diameters and comparison was made with SWBXT measurements of Burgers vectors, Frank's relationship between the hollow-core diameter and the Burgers vectors of the micropipes [4] was found to hold [5]. SWBXT has also revealed the presence of basal plane dislocations in 4H and 6H SiC. The configurations of these dislocations suggest they are generated as a result of thermal stresses during post-growth cooling and SWBXT shows they are associated with micropipes; under the electron microscope they can be seen to terminate at micropipe walls [6]. SWBXT has shown the presence of small-angle boundaries, notably in 4H-SiC [7]. While the densities of hollow-core screw dislocations, which have been shown to be most detrimental to device performance, have been drastically reduced, closed-core screw dislocations and basal plane dislocations persist in appreciable densities. Their

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characterization is very important both to help crystal growers develop strategies for their minimization or elimination and to aid in the understanding of the influence of these defects on device performance. SWBXT is well suited to this task. The work of Neudeck et al [8-10], highlights the significant effect closed-core screw dislocations can have on device performance.

SWBXT has also played a strong role in the characterization of epilayers grown on SiC substrates [11]. For the case of 6H homo-epitaxy, substrate screw dislocations which threaded the growth surface, were found, as expected, to be replicated in the epilayer, However, basal plane dislocations found in the substrate were observed to be mostly absent from the epilayer. The absence of basal plane dislocation generation was attributed to the lower growth temperatures of the CVD-grown films. The simultaneous structural and microstructural capability of SWBXT has been found to be indispensable for the characterization of 3C on 4H hetero-epitaxial layers [12-15]. SWBXT is capable of distinguishing between the two possible 3C variants which can nucleate during deposition, while simultaneously yielding information on defect content and distortion. It also revealed the presence of mismatch strain between the 3C and 4H. This mismatch was investigated further with High Resolution Triple-Axis X-ray Diffraction (HRTXD) and the results were correlated.

In this paper, we will provide an overview of the applications of SWBXT and HRTXD to: (1) the analysis of defects in SiC substrates, and (2) the study of 3C/4H hetero-epitaxial layers. The particular strengths of the techniques in each application will be highlighted and illustrated.

SWBXT Characterization of SiC Substrates

Synchrotron topography comprises a set of techniques capable of revealing the distribution of strains and defects in large single crystal materials. White Radiation Topography [16] is basically analogous to the Laue Transmission technique, except with greatly enhanced capabilities which derive from the natural collimation and high intensity of the synchrotron beam. Long beamlines lead to small angles subtended by the source at points in the specimen, which in turn leads to excellent geometrical resolution capabilities. Thus, in a synchrotron source a large area X-ray beam, with inherent low divergence, and with excellent geometrical resolution capability.

An SWBXT image recorded in back-reflection geometry from a commercially available (0001) 6H SiC crystal is presented in Fig. 1(a). It has been previously demonstrated via simulation that the screw dislocation images, formed largely through orientation contrast, consist of black rings surrounding white circles, with the diameter of the rings being roughly proportional to the Burgers vector. From such images, the distribution of the micropipes and screw dislocations as well as their detailed structures can be obtained. By using a scanning device, SiC wafers with diameters up to several inches can be nondestructively imaged by SWBXT. Moreover, SiC wafers with devices fabricated on them can be imaged, further emphasizing the advantages of this technique. Images of such screw dislocations recorded from wafers cut parallel to the growth-axis, and their successful simulation, confirm their nature and the fact that they are growth dislocations. A transmission image recorded from the same crystal shown in Fig. 1(a) is shown in Fig. 1(b). Note the one-to-one correspondence between the screw dislocation images (some examples are indicated by arrows). Detailed analysis of these images has enabled the equations for the screw dislocation strain field to be confirmed. The transmission image also reveals the presence of networks of basal plane dislocations. The morphology of these networks, often in the form of nearly concentric loops emanating from the crystal edges or from micropipes, is clearly associated with a deformation process, possibly occurring during post-growth cooling. Similar images are obtained from 4H-SiC.

Characterization of 3C/4H Hetero-Epitaxial Layers

SWBXT has been successfully used to identify the polytype distribution in hetero-epitaxial layers of 3C grown on 4H [10-15]. In this research, the 4H substrate is divided up into an array of mesas by etching suitable trenches, and then the mesas are subjected to a pure stepflow homoepitaxy procedure designed to “grow out” all atomic steps prior to the heteroepitaxial process [17]. This is important since the presence of atomic steps on the mesa means that different termination stacking sequences will be present side by side on the same mesa. Therefore, if 2D nucleation of 3C occurs on mesas with steps, more than one 3C variant is likely nucleate on a given mesa. However, the removal of atomic steps is only possible in mesas free from threading screw dislocations which are a continual source of steps. Thus, if the heteroepitiaxial growth is initialed on mesas rendered step-free by the pre-heteronucleation procedure, mesas which are free from threading screw dislocations should experience uniform nucleation of a single 3C variant, while other mesas (not step-free) are likely to experience simultaneous nucleation of both variants. SWBXT, with its simultaneous structural and microstructural capability, is ideally suited to provide maps of such polytype distributions. Detailed mapping of the distribution of the polytypes and their variants was carried out using SWBXT, in the back-reflection geometry in the case of 4H, and in grazing-incidence reflection geometry in the cases of 3C(I) and 3C(II) [12, 13]. The information provided by the reflections recorded in these two geometries were combined by image processing, resulting in a single figure showing the distribution of the 3C variants and the positions of the screw dislocations across the sample [15]. Fig. 2(a) shows an image that is made up of the superimposition of the three topographs described above, image processed so that they all are referred to the same exact scale. For such 3C/4H crystals, back reflection topographs revealed strain contrast bounding regions that had experienced 3C growth. Transmission topography enabled the directionality of the displacement field associated with the strain field to be determined. This displacement was found to be oriented perpendicular to the edges of the 3C regions, with the contrast disappearing when the reflection vector projected parallel to an edge of a 3C region.

The crystalline quality of the 3C epilayer on the 4H mesa substrate was further investigated using HRTXD. The diffractometer employed was a Bede D1 system and the X-ray beam was limited to be 1 or 2 mm in diameter by pinholes. The marked mesa in Fig. 2(a) is a 0.4×0.4 mm^2 terrace fully covered by a single-variant 3C epilayer, as indicated by the pure blue color. Fig. 2(b) is the θ-scan rocking curve taken from this mesa. The full width at half maximum (FWHM) values of the 0004_{4H} and 111_{3C} peaks are only 15.6 and 17.3 arcsec, respectively, indicating that the crystalline quality of both the heteroepilayer and substrate is very high. The inset is the corresponding triple-axis reciprocal space map, in which the well-shaped diffraction spot of the 3C epilayer shows no detectable lattice misorientation or d-spacing fluctuation. Furthermore, the perfect alignment of the two diffraction spots along the Q(0004) direction shows that the $(111)_{3C}$ and $(0001)_{4H}$ planes are perfectly parallel. This is consistent with the 2D nucleation mechanism of 3C growth [14].

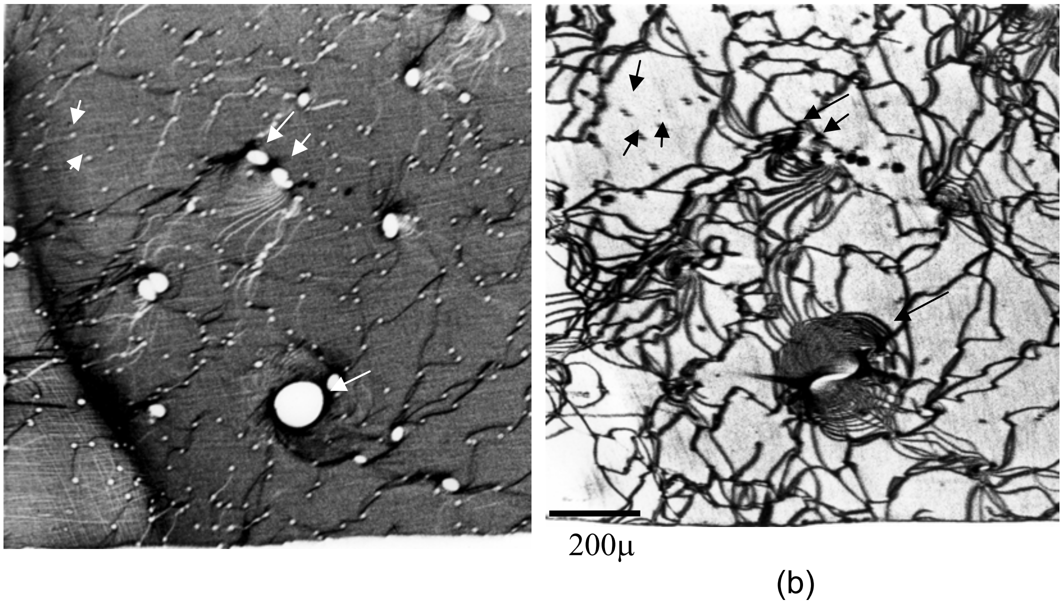
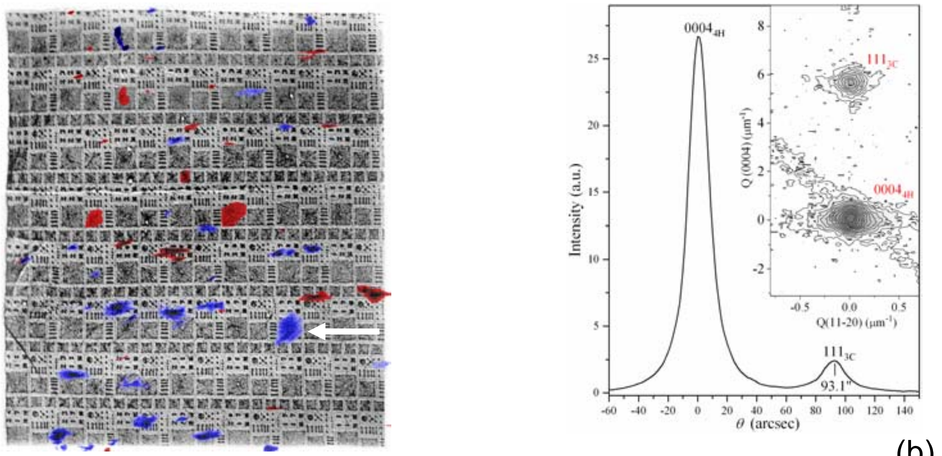


Fig. 1. (a) Back-reflection SWBXT image recorded from a 6H-SiC wafer thinned to around 30 μm. The circular images (black rings surrounding white circles) correspond to screw dislocation images; (b) transmission image recorded from the same region of crystal.

Although the out-of-plane mismatch can be readily calculated to be $\Delta c/c \approx -0.14\%$ ($c_{4H} > c_{3C}$) from the symmetric-reflection rocking curve in Fig. 2(b), the measurement of the in-plane mismatch for 3C/4H heterostructure is difficult due to the different symmetries of 3C and 4H crystals. To overcome this difficulty, we have recently developed a novel diffraction method for absolute lattice parameter measurements with a precision of $10^{-6} \sim 10^{-7}$ Å [17]. In this method, one only needs to measure the angular difference Θ between the 0004_{4H} (111_{3C}) and 0008_{4H} (222_{3C}) reflections to obtain the c parameter. By measuring another parameter Θ_A, the angular difference between 0004_{4H} (111_{3C}) and $11\bar{2}8_{4H}$ (402_{3C}), one can then obtain the a parameter. Thus, we have simultaneously but independently derived the lattice parameters for 3C and 4H: $c_{3C} = 2.517586$, $a_{3C} = 3.08039$, $c_{4H} = 2.521119$, $a_{4H} = 3.07950$ Å. The two mismatch parameters are then: $\Delta c/c = -0.001401$ and $\Delta a/a = 0.000289$. Apparently, a_{3C}/c_{3C} ratio is slightly smaller than that of a cubic structure ($\sqrt{3/2}$), indicating the epilayer is partially relaxed.



(a) (b)

Fig. 2 (a) Composite image showing the distribution of 3C(I) (red) and 3C(II) (blue) in the heteroepitaxial structure; (b) 0004 rocking curve of the arrowed mesa in (a). $CuK\alpha_1$ radiation. Inset is the triple-axis reciprocal space map, in which the inclined streak on 0004_{4H} is the analyzer streak.

Conclusions

SWBXT is an extremely useful technique for the characterization of defects in SiC crystals. To date it has provided complete quantitative characterization of both closed-core and hollow-core screw dislocations and basal plane dislocations, and has also provided insight into the formation mechanisms of these defects. Being capable of imaging defects in wafers with devices fabricated on them, it has also enabled much light to be shed on the influence of defects on device performance. It has also enabled polytype mapping in 3C/4H heterostructures facilitating optimization of growth conditions.

Acknowledgments

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