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X-ray topographical analysis of 4H-SiC epitaxial layers using a forward-transmitted beam under a multiple-beam diffraction condition

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Multiple-beam diffraction X-ray topography was used to determine the Burgers vector **b** of threading edge dislocations (TEDs) and basal plane dislocations (BPDs) in 4H-SiC epitaxial layers. In hexagonal crystals, the technique simultaneously yields five different diffractions corresponding to different diffraction vectors **g**. Hence, this method enables us to determine the components of **b** using the $\mathbf{g} \cdot \mathbf{b} = 0$ rule without widely changing the diffraction geometry. The **b** vectors of TEDs and BPDs were successfully determined by the method. These results were then confirmed by ordinary grazing-incidence X-ray topography in order to verify the validity of this technique. © 2018 The Japan Society of Applied Physics

4H silicon carbide (4H-SiC) holds considerable promise as a next generation semiconductor material due to its superior material properties, which include a high breakdown field, high thermal conductivity and a wide band gap.¹⁻³ Recently, 4H-SiC power electronic devices (diodes and MOSFETs) are used in low-loss power controls in various industries, home appliances, automobiles and trains.⁴ However, 4H-SiC epitaxial layers (epilayers) still contain many dislocations such as threading screw dislocations (TSDs), threading edge dislocations (TEDs) and basal plane dislocations (BPDs),⁵ which can degrade the performance of the devices.^{6,7)} To improve device performance and reliability, it is important to distinguish dislocation types^{8–20)} and investigate their electrical effects on devices.^{21–23)}

The KOH etching method is widely used to determine the locations and kinds of dislocations in 4H-SiC epilayers.²⁴⁾ However, this method cannot evaluate the directions of Burgers vectors **b** of dislocations. X-ray topography enables us to determine the directions of **b** vector by using ordinary $\mathbf{g} \cdot \mathbf{b}$ analysis,²⁵⁾ where \mathbf{g} is a diffraction vector. However, the ordinary method requires different diffraction geometries among which sample rotations at least 60° (or 120°) and fine adjustments are inevitable. In addition, since the topographical images thus obtained are differently deformed among different diffractions, image processing to recover the original shape is required for the $\mathbf{g} \cdot \mathbf{b}$ analysis.

The multiple-beam diffraction method can eliminate these troublesome procedures. This method simultaneously yields different diffractions corresponding to different g vectors. We can hence perform the $g \cdot b$ analysis without widely changing a diffraction geometry. In addition, since a forward-transmitted beam is used, topographs obtained are not deformed from the original. Multiple-beam diffraction X-ray topography was previously applied for a sapphire and silicon wafer.²⁶

In this study, we determined the b vectors of TEDs and BPDs in 4H-SiC epilayers using the multiple-beam diffraction method. The b vector directions were also determined by ordinary grazing-incidence X-ray topography to prove the validity of the multiple-beam diffraction method.

The samples examined were 4°- and 8°-off-cut (0001) Si-face 4H-SiC wafers with 160- and 130- μ m-thick epilayers, respectively. Substrates were removed from the rear side by polishing to obtain self-standing samples for diffraction measurements. The final thicknesses of 4°- and 8°-off samples, were 150 and 110 μ m, respectively. Specular surfaces were prepared on the front side surface by chemical mechanical polishing (CMP).

Synchrotron X-ray grazing-incidence topography was performed at SPring-8 BL08B2 under the condition of $g = 11\overline{2}8$ with $\lambda = 0.146$ and 0.154 nm (8.52 and 8.05 keV) for 4°-and 8°-off samples, respectively. Grazing-incidence condition was given by an asymmetric reflection, where the incident beam angle was adjusted to 5° from the sample surface.⁹

Multiple-beam diffraction X-ray topography was carried out at SPring-8 BL24XU utilizing a monochromatic X-ray beam at $\lambda = 0.0827$ nm (15 keV). The measurement geometry is shown in Fig. 1(a). The X-ray beam was adjusted to the size of $1.0 \times 1.0 \text{ mm}^2$, monochromatized, and then incident on a SiC sample placed on a goniometer. The transmitted beam was received by a scintillator coupled with a relay lens optics and CMOS camera, while the diffracted beams were received by a fluorescent screen. Note that each of the beams accompanies a topography image of about $1.0 \times 1.0 \text{ mm}^2$, where the forward transmitted and diffracted beams are complementary (a positive image and negative images are observable, respectively). Unlike ordinary X-ray topography, the multiple beam method uses the forward-transmitted image, which is visualized by the CMOS camera, instead of the diffracted images. Figure 1(a) shows the multiplebeam diffraction condition, in which the incident beam separates in one transmitted and five diffracted beams. In this case, 2200, 2110, 1210, 1010, and 0110 diffraction vectors are located within the basal plane and meet the following relations: $g\bar{2}200 = g\bar{1}2\bar{1}0 + g\bar{1}010 = g\bar{2}110 + g01\bar{1}0$ as shown in Fig. 1(b). Accordingly, the five diffractions; $\overline{2}200$, $\overline{2}110$, $\overline{1}2\overline{1}0$, $\overline{1}010$, and $01\overline{1}0$ are simultaneously obtained.²⁶⁾

When the simultaneous diffraction occur, dislocations with every **b** vectors are visible. However, for example, maintaining only the $01\overline{10}$ diffraction condition, we can slightly rotate the sample by about 0.1° around the axis parallel to the



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Fig. 1. (Color online) (a) Schematic drawing of an experimental setting of forward-transmitted topography. (b) Schematic drawing of geometry with five diffraction vectors $g = \bar{2}200$, $\bar{2}110$, $\bar{1}2\bar{1}0$, $\bar{1}010$, and $01\bar{1}0$ in multiple-beam diffraction conditions.

[0110] direction. This can let the $\overline{2}200$, $\overline{2}110$, $\overline{1}2\overline{1}0$, and $\overline{1}010$ diffractions be out of diffraction conditions. Thus, only dislocations having **b** vectors perpendicular to the 0110 direction disappear, while other dislocations retain contrasts. In the same manner, each of $\overline{2}200$, $\overline{2}110$, $\overline{1}2\overline{1}0$, and $\overline{1}010$ diffraction conditions can be realized by rotating the sample about 0.1° around each **g** vector direction. TED and BPD in 4H-SiC have $\mathbf{b} = a/3(11\overline{2}0)$, while **g** vectors parallel to the m directions are required to distinguish the **b** vectors. This multiple-beam diffraction condition contains three **g** vectors parallel to the m direction, making it suitable to analyze 4H-SiC materials and unlike the previous setting containing two **g** vectors parallel to the m direction.²⁶

The grazing-incidence X-ray topography (g = 1128) was carried out for the 8°-off epilayer as shown in Fig. 2(a), where many dislocation contrasts can be observed. The bright long shape contrast with a dark tail within a long rectangle represents a BPD parallel to the step-flow direction. A bright, large circular contrast shows a TSD approximately normal to the surface, while the other point contrasts represent TEDs. It is known that BPDs that propagate into epilayers have b vectors parallel or antiparallel to the step-flow direction.²⁷⁾ Hence, the **b** vector of the observed BPD is expected to be $[11\overline{2}0]$ or $[\overline{1}\overline{1}20]$. Images of TEDs exhibit six different shapes corresponding to the six directions of extra halfplanes, and thus allowing us to distinguish the directions of TEDs' **b** vectors.⁹⁾ Figure 2(b) shows the map of TEDs in the identical area of Fig. 2(a). The six types of TED are drawn by "T-marks" with six colors indicating the directions of the extra half planes in the inset of Fig. 2(b), where the direc-



Fig. 2. (Color online) (a) Synchrotron X-ray grazing incidence topographic image ($g = 11\overline{2}8$) of the 8°-off 4H-SiC epilayer. Red, green, blue, and white arrows indicate TEDs with different **b** vectors. (b) Schematic drawing of the dislocation map of the identical area of (a). The six types of the TEDs are drawn by "T-marks" showing their extra half plane directions. The directions of **b** vectors are also shown in the inset with six colors, while four of the six TED types are observed in the area.

tions of the b vector are also shown. Four of six types of TED are observed in the area of Fig. 2(a), where they are indicated by red, green, blue, and white arrows.

The forward-transmitted X-ray topography under multiplebeam diffraction was carried out for the same sample as shown in Fig. 3(a). The BPD shows long linear contrast along the step-flow direction. TEDs show short linear segments corresponding to transmission images of inclined dislocations penetrating from the front to rear surfaces of the sample. Since all five diffractions were given under this condition, all TEDs in the area have topographical contrasts. For example, four types of TEDs indicated by white rectangles marked by "W", "G", "R", and "B" are all visible in Fig. 3(a).

Figure 3(b) shows the forward-transmitted X-ray topographic image in $01\overline{1}0$ diffraction, where only $01\overline{1}0$ diffraction is maintained, and others are outside the condition. In this figure, the contrast of the TED in a rectangle "W" disappears, while those in "G", "R", and "B" retain dislocation contrasts. This means that the TED in a rectangle "W" has **b** vector perpendicular to the **g** vector; that is, $\mathbf{g} \cdot \mathbf{b} = 0$ is satisfied for $\mathbf{g} = [01\overline{1}0]$.

Figure 3(c) shows the topographic image in 1010 diffraction, where the contrast of the TED in the rectangle "G" seems disappeared. Although faint contrast is visible near the dislocation edges at the front and rear side surface, it can be regarded that $\mathbf{g} \cdot \mathbf{b} = 0$ is satisfied for this TED.

Since the $\overline{2}200$ diffraction was not sufficiently strong, the $\overline{1}100$ diffraction topography was employed instead as shown in Fig. 3(d). Both $\overline{1}100$ and $\overline{2}200$ diffractions have the same effect for $\boldsymbol{g} \cdot \boldsymbol{b}$ analysis of TEDs. In this figure, the BPD in a long rectangle and the TEDs in rectangles with "R" and "B" are out of contrast in Fig. 3(d). Hence, the \boldsymbol{b} vector directions of the BPD and TEDs are perpendicular to the diffraction vector, and $\boldsymbol{g} \cdot \boldsymbol{b} = 0$ condition is satisfied for the BPD and TEDs marked by "R" and "B".

Figure 4 summarizes the results of the $g \cdot b$ analyses based on the grazing-incidence topography shown in Fig. 2 and the



Fig. 3. (Color online) (a) An image of forward-transmitted X-ray topography under multiple-beam diffraction condition. White, green, red, and blue arrows indicate TEDs, which show linear segments corresponding to transmission images of inclined dislocations penetrating from the front to rear surfaces of the sample. A BPD is included in the long rectangle, and TEDs labeled "W", "G", "R", and "B" locate in the four short rectangles. Panel (b) shows a forward-transmitted topography image in 0110 diffraction. The contrast of the "W"-type TEDs disappears under this condition. Panel (c) shows a topography image in 1010 diffraction. The contrast of "G"-type TEDs disappears under this condition. Schematic drawings of "T-marks" and Burgers vectors of the "White", "Black", "Green", "Yellow", "Red", and "Blue"-type TEDs are shown in the insets.

forward-transmitted topography shown in Figs. 3(a)-3(d). The \boldsymbol{b} vector directions given by the grazing-incidence X-ray topography are listed for the four TEDs ("W", "G", "R", and "B") and the BPD in the third row. The forward-transmitted images with three g vectors are provided from fourth to sixth rows for the five dislocations. The circles (\bigcirc) or crosses (\times) indicate that dislocation images are visible or invisible, respectively. As shown here, the invisibility criterion of four types of TEDs is well consistent with the assignment of the *b* vectors (in third row) determined by the grazing-incidence topography. For examples, the TED "W" having $b = [2\overline{1}\overline{1}0]$ gives invisible contrast for $g = 01\overline{10}$, where $g \cdot b = 0$ is satisfied. Similarly, the TED "G" with $\boldsymbol{b} = [\bar{1}2\bar{1}0]$ shows invisible contrast for $g = \overline{1010}$. The images of the TEDs "R" and "B" with $\boldsymbol{b} = [11\overline{2}0]$ and $[\overline{1}\overline{1}20]$, respectively, disappear in $g = \overline{1}100$ diffraction. In addition, yellow- and black-type TED were also confirmed in other area. Also for the BPD with $\boldsymbol{b} = [11\bar{2}0]$ or $[\bar{1}\bar{1}20]$, the dislocation image disappears for $g = \overline{1}100$, showing that the **b** and **g** vectors are perpendicular to each other. We can thus confirm that the b vector directions determined by the multiple-beam X-ray diffraction method are consistent with those estimated by the ordinary method already established.

From the results above, the multiple-beam X-ray diffraction method exhibits no shape deformation, nor any position shift of the dislocation lines on the topographs [Figs. 3(a)-3(d) and its validity of **b** vector determination. Recently, the impact on dislocation features exerted by \boldsymbol{b} vector directions becomes apparent in 4H-SiC. For example, it was elucidated that the TEDs extended with unique inclination angles and directions toward the step-flow direction in the epilayer depending on their extra half plane (**b** vector) directions.¹⁰⁾ It also became apparent that BPDs in pn devices transformed into triangular or bar-shaped stacking faults depending on their b vector directions by forward current operation or UV irradiation.^{20,28)} Considering evaluations of such 4H-SiC epilayers with dislocation densities of 10³ cm⁻², by conventional topography, individual consecutive collation of dislocations using several topographs taken by different gvectors is supposed to be difficult, because the relative position of many dislocations changes based on the projection directions of different g vectors. Although the multiple-beam X-ray diffraction method can only measure an area of 1×1 mm^2 , it can take several topographs with different g vectors at once. A wider-areal evaluation is effective by measuring repetitively with X-Y movement for 4H-SiC epilayers and other materials.



(o: visible, x: invisible)

Fig. 4. (Color online) Collation of the results between the grazing-incidence X-ray topography and the forward transmitted X-ray topography.

In conclusion, multiple-beam diffraction X-ray topography method was investigated to analyze b vectors of threading dislocations in 4H-SiC. This method can conduct the $g \cdot b$ analysis without widely changing a diffraction geometry. Using this method, we successfully determined the directions of b vectors of TEDs and BPDs in 4H-SiC. The b vectors thus determined were confirmed correct by the ordinary grazing-incident X-ray topography.

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