Synchrotron X-ray Topographic Study on Nature of Threading Mixed Dislocations in 4H-SiC Crystals Grown by PVT Method

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Abstract

Synchrotron X-ray Topography is a powerful technique to study defects structures particularly dislocation configurations in single crystals. Complementing this technique with geometrical and contrast analysis can enhance the efficiency of quantitatively characterizing defects. In this study, the use of Synchrotron White Beam X-ray Topography (SWBXT) to determine the line directions of threading dislocations in 4H-SiC axial slices (sample cut parallel to the growth axis from the boule) is demonstrated. This technique is based on the fact that the projected line directions of dislocations on different reflections are different. Another technique

also discussed is the determination of the absolute Burgers vectors of threading mixed dislocations (TMDs) using Synchrotron Monochromatic Beam X-ray Topography (SMBXT). This technique utilizes the fact that the contrast from TMDs varies on SMBXT images as their Burgers vectors change. By comparing observed contrast with the contrast from threading dislocations provided by Ray Tracing Simulations, the Burgers vectors can be determined. Thereafter the distribution of TMDs with different Burgers vectors across the wafer is mapped and investigated.

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Introduction

Threading screw dislocations (TSDs) have long been observed in 4H-SiC crystals grown by Physical Vapor Transport (PVT) method. It has been reported that the density of TSDs has an impact on the reverse characteristics such as leakage current and breakdown voltage for certain types of SiC power devices [1-4]. Recent study [5] shows that a significant amount of the threading dislocations with screw component are actually mixed type dislocations with Burgers vectors $nc + ma$ (where m and n are integers). These dislocations are called Threading Mixed Dislocations (TMDs). TMDs have both edge (“a”) and screw (“c”) components. TMDs are either inherited from the seed crystals or nucleated as opposite-sign pairs at the seed interface in the early stage of crystal growth or other nucleation sites during later stages. The existence of voids or inclusions during growth can lead to the formation of threading dislocations. And the

character of the dislocations nucleated depends on the nature of the lattice closure failures induced by the voids or inclusions. In this study, we will demonstrate how to determine the Burgers vectors and line directions of these TMDs using Synchrotron X-ray Topography analysis. Ray tracing simulation was used here to simulate the contrast from TMDs on a SMBXT image. The simulation results provide an unambiguous method to determine the exact Burgers vectors of TMDs in commercial offcut wafers. Spatial distribution of both the edge and screw component of the TMDs as well as the dislocation character distribution across the wafer is investigated.

Experiments

Synchrotron X-ray Topography work was carried out at Beamline 1-BM, Advanced Photon Source (APS) in Argonne National Lab (ANL). Transmission images were obtained from a 4H-SiC axial slices with diffraction vector $(\bar{1}2\bar{1}0)$ and $(1\bar{1}00)$ using white beam radiation. Grazing-incidence images were recorded from a commercial 100mm 4H-SiC wafer using monochromatic beam (8.99KeV) with 2° grazing angle. The diffraction vectors are $11\bar{2}8$, $2\bar{1}18$, $\bar{1}218$, $\bar{1}128$, $\bar{2}118$ and $\bar{1}2\bar{1}8$. The sample-film distance is around 30cm. Crystals were sent by Cree. Ray tracing simulation was carried out to provide contrast from TMDs with different Burgers vectors on topographs with different diffraction vectors.

Results and Discussions

1. Line direction determination

It is important to study the line directions of dislocations in 4H-SiC. In the case of basal plane dislocations (BPDs) which are induced by stress, the line directions

provide information on the active slip systems and the stress state on the slip planes. The growth dislocations such as threading dislocations usually adopt low energy directions in the lattice. Determination of line directions of these growth dislocations provides information on their formation mechanism during growth. Threading edge dislocations (TEDs), particularly, were observed to glide in the prismatic slip system during growth. The determination of the line directions of TEDs in this case is also of great importance since it provides information about the radial stress state during PVT growth. SWBXT is very helpful in determining the line directions of dislocations in single crystals. White beam spectrum gives multiple reflections simultaneously, on each of which dislocation lines are present with a different projected directions. Measuring the projected line directions on two different reflections can provide us the 3-dimensional line direction of the dislocation in the crystal. Fig. 1 shows the basic idea of this technique.

A dislocation is present in the crystal with line direction along vector $\mathbf{l}$ and two different reflections are recorded on the recording plate. In reflection #1, the projected line of the dislocation A is along $\mathbf{l}_1$ direction. The position of spot #1 is identified by the diffracted beam wave vector $\mathbf{s}_{g_1}$. So basically $\mathbf{l}_1$ and $\mathbf{s}_{g_1}$ make up a plane with normal $\mathbf{n}_1$. Similar for spot #2, a plane consisting of $\mathbf{l}_2$ and $\mathbf{s}_{g_2}$ can be identified by the plane normal $\mathbf{n}_2$. These two plane normal are given by

$$\mathbf{n}_1 = \mathbf{s}_{g_1} \times \mathbf{l}_1, \mathbf{n}_2 = \mathbf{s}_{g_2} \times \mathbf{l}_2$$

Therefore the line direction of dislocation A in real crystal is simply given by the cross product of the two plane normals

$$\mathbf{l} = \mathbf{n}_1 \times \mathbf{n}_2$$

The actual computation process can be also done by a simple compute program, in which the structure profile and orientation of the crystal have to be identified

beforehand including lattice parameters, surface plane, side plane orientations as well as the exact orientation of the samples in actual experiments. Then measurements have to be made on actual topographs to provide the projected line directions of the same dislocation on two different reflections. The rest of the calculation process will be done by the program.

A demonstration of this method is given here. In this case, a 4H-SiC axial slice with $(11\bar{2}0)$ surface orientation was used. The sample was placed in a way that the surface normal was at 16° with respect to the incident beam so that the $\bar{1}2\bar{1}0$ and $1\bar{1}00$ reflections could be obtained simultaneously on the either side of the direct beam. Recording plate was placed 20cm away from the sample and was perpendicular to the beam incident direction. The topographic images of these two reflections are shown in Fig. 2(a). Dislocation of interest is marked with arrow.

The $\mathbf{g} \cdot \mathbf{b}$ analysis was carried out on this dislocation using other reflections and it was confirmed that this dislocation is a TMD. As we can see, the dislocation on $\bar{1}2\bar{1}0$ reflection is obviously tilted with respect to the vertical axis, which in this case is the c axis, while the dislocation image on $1\bar{1}00$ reflection is almost parallel to the growth axis. Measurements show that l_1 is 4° off the vertical axis on the recording plate and l_2 is 12° off the vertical axis. The Cartesian coordinate system is set up in a way that the origin is at the core of the dislocation of interest, x axis points to $[11\bar{2}0]$ crystalline direction, y axis points to $[\bar{1}100]$ direction and z axis points to the $[0001]$ direction. In this way, the incident beam direction $\mathbf{s}_0$, the diffracted beam directions $\mathbf{s}_g$ and the projected line directions $\mathbf{l}_1$ and $\mathbf{l}_2$ on different reflections can be expressed as vectors with 3-index coordinates. The coordinates for the actual line direction $\mathbf{l}$ can be therefore calculated. The calculation result is then visualized with schematic

drawing and shown in Fig. 2(b). The dislocation is roughly parallel to the c axis (taking a small angle 3.7° to c axis). And it is lying on a crystalline plane that is 22.8° with respect to the $(1\bar{1}00)$ prismatic plane.

This technique is very useful for quickly determining the line directions of threading dislocations in axially-cut sample. The accuracy of the measurements is mainly dependent on the accuracy of measuring sample orientation in the imaging process, as well as the accuracy of measuring projected line directions on the topographs. To ensure the sample orientation is precisely measured, the Laue pattern is often recorded with exactly the same setup before or after imaging, since the sample orientation can be easily and accurately obtained through Laue patterns. This technique works well for growth dislocations including TMDs and TEDs and can readily be applied to any other single crystal such as GaN, AlN, sapphire and other technologically important materials.

II. Burgers vector determination

Burgers vector is another important property of a dislocation. The determination of Burgers vectors of growth dislocations such as TSDs, TMDs in the crystals provides useful information regarding their formation mechanisms during growth. Spatial distribution of these threading dislocations with different Burgers vectors indicates the stress state distribution during growth especially the early stage of growth. TMD has been reported [5] to be the predominant type of threading dislocation with screw component in 4H-SiC crystals grown by PVT method. Ray tracing simulation is used here to simulate the contrast from TMDs on grazing topographs with different diffraction vector $\mathbf{g}$. This method is based on the fact that orientation contrast is the predominant contributor to contrast from a dislocation in back-reflection and grazing incidence geometries [6]. The

mechanism of ray tracing simulation in grazing-incidence setup is shown in Fig. 3, in which a threading dislocation with Burgers vector $\mathbf{b}$, is present at the origin. It is associated with a displacement field around its core. The displacement field $\mathbf{u}$ is the quantified amount of strain that the dislocation brings to the crystal in the vicinity. The normal of the distorted reflecting planes $\mathbf{n}$ can be given by

$$\mathbf{n} = \mathbf{n}_0 - \nabla(\mathbf{n}_0 \cdot \mathbf{u})$$

where $\mathbf{n}_0$ is the plane normal of perfect reflecting planes. When X-rays are incident (with incidence wave vector $\mathbf{s}_0$) on the crystal surface, local distortion on the reflecting planes ($\{11\bar{2}8\}$ planes in this case) will result in the variation of diffracted beam directions $\mathbf{s}_g$. The diffracted beam direction is given by

$$\mathbf{s}_0 \times \mathbf{n} = -\mathbf{n} \times \mathbf{s}_g$$

Diffracted beams with slightly different directions will be collected on the recording plane and contrast is therefore formed as the diffracted beams are overlapping or separated.

Ray tracing simulation shows that on $11\bar{2}8$ type grazing-incidence topographs the contrast from a TMD varies with the relative angle between the Burgers vector of its edge component and the diffraction vector associated with the topograph.

For TMDs with positive screw component ($+c$ or $[0001]$), when this angle equals to 120° or 180° , the contrast will have a significant difference: the elliptical white center narrows while the dark contrast edge does not completely enclose it. And for TMDs with negative screw component ($-c$ or $[000\bar{1}]$), when this angle equals to 0° or 60° , similar contrast features will be observed. This is shown in Fig. 4(a). The rapid way to determine the signs of the screw components is to look at the elliptical white center. If it is canted clockwise, the screw component is along $+c$ and if counter-clockwise, it will be $-c$. Real grazing

topographs recorded using synchrotron monochromatic beam are shown in Fig. 4(b) and confirm the simulation results. Therefore the Burgers vector of any TMD can be determined by recording six different $11\bar{2}8$ grazing topographs and then analyzing the contrast change with the diffraction vector. For the case of TMDs with Burgers vectors " $c + na$ " where n is larger than 1, simulation results (not shown here) confirm that their contrast follows the same pattern as " $c + a$ " case and is actually more distinguished from TSDs. Therefore this method also applies for these " $c + na$ " TMDs.

In order to get the spatial distribution of the Burgers vectors of TMDs across the wafer, five different regions are selected from a 100mm commercial 4H-SiC wafer. Each region is 5mm by 5mm in size. SMBXT work was carried out on all five regions and the Burgers vectors of the TMDs within each region were determined. The result is shown in Fig. 5 where the approximate locations of these five regions are illustrated. Three types of statistical work have been carried out on each region. First of all, the percentage of the TMDs over the sum of TMDs and TSDs is given here. It is consistent with other studies [5] in that in 4H-SiC grown by PVT, most threading dislocations with screw component are TMDs. The next result shows the distribution of the screw components of TMDs and TSDs across the wafer. It is obvious that the regions away from the major cut is dominated by $-c$ type threading dislocations and this predominance is reduced along $[\bar{1}100]$ direction. Although many of the threading dislocation were replicated from the seed during growth, this might still indicate the seed interface condition is not uniformly distributed across the area of the wafer, leading to the nucleation of more threading dislocations with negative screw component on one end of the crystal than the other end. As for the edge components associated with the TMDs, analysis has been done as well to reveal their spatial distribution across

the wafer. For easier demonstration, six types of edge component Burgers vectors are labeled “ $0^\circ a$ ”, “ $60^\circ a$ ”, “ $120^\circ a$ ”, “ $180^\circ a$ ”, “ $240^\circ a$ ” and “ $300^\circ a$ ” based on their relative angle with respect to $1/3[11\bar{2}0]$. The percentages of all type edge components for each region are demonstrated. It is clear that these six types Burgers vectors are not, again, uniformly distributed across the wafer. In region #1, #2 and #4, one certain type is dominating, where one of the screw component also dominates. Furthermore, the TMD density in these three regions is also comparably higher than the other two. It is likely that nucleation of TMDs was occurring during growth and the nature of the nucleation sites (most likely inclusions or voids sites) favors the adoption of certain edge component into the dislocation nucleation process.

Conclusions

This paper introduces two techniques to determine the line directions and Burgers vectors of threading dislocations in 4H-SiC crystals grown by the PVT method using Synchrotron X-ray Topography. The determination of line directions of TMDs in axial slices requires the transmission SWBXT images on two different reflections. The line directions can be obtained by measuring the projected line directions of dislocations of interest on these two reflection. The determination of Burgers vectors of TMDs in commercial SiC off-cut wafers requires the all six types of $11\bar{2}8$ grazing-incidence images recorded using synchrotron monochromatic beams. By comparing the contrast of the dislocations on these six reflections and looking for the two reflections with significantly different contrast, we will be able to unambiguously determine the exact Burgers vectors of the TMDs in the wafer. Further investigation on the spatial distribution of the different types of TMDs indicates that TMDs have nucleated during crystal

growth and the seed interface conditions were not uniformly distributed across the wafer.

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Fig.1 Schematics showing the mechanism of this technique for determination of line directions of dislocations

Fig. 2 (a) Transmission topographs recorded by synchrotron white beam radiation from a 4H-SiC axial slice. Two reflections are presented here. Dislocation of interest is marked with red arrow. (c) Visualization of the determined line direction of the dislocation of interest. The dislocation is almost parallel to the c axis.

Fig. 3 The mechanism of ray tracing simulation for grazing-incidence geometry

Fig. 4 (a) Simulated images of a TMD with Burgers vector $1/3[11\bar{2}3]$. On the two reflections marked with red box, the contrast shows significant difference.

The relative angles between the diffraction vectors of these two reflections and the Burgers vector of edge component are 0° and 60° respectively.

(b) Actual topographic observation of a TMD on six $11\bar{2}8$ reflections. The screw component of this TMD is determined to be $-c$ since the white center is apparently canted counter-clockwise with respect to the diffraction vector. Then, the contrast on the first two reflections shows significant difference. By comparing with the simulated images, the Burger vector of the edge component is determined to be $1/3[11\bar{2}0]$. Therefore the Burgers vector of this TMD is determined to be $1/3[11\bar{2}3]$.

Fig. 5 Results showing the spatial distribution of the characters and Burgers vectors of TMDs across the wafer. Three types of statistical work have been carried out on each region: TMD percentage, c/-c screw component ratio, six type edge components ratio. The results for each region are shown as three rows of colorful boxes. Refer to the explanatory notes for detailed correspondence.