ARTICLE IN PRESS

Journal of Crystal Growth ■ (■■■■) ■■■–■■■

Contents lists available at ScienceDirect

Journal of Crystal Growth

journal homepage: www.elsevier.com/locate/jcrysgro

Synchrotron X-ray topographic study on nature of threading mixed dislocations in 4H–SiC crystals grown by PVT method

Jianqiu Guo [a,*], Yu Yang [a], Fangzhen Wu [a], Joe Sumakeris [b], Robert Leonard [b], Ouloide Goue [a], Balaji Raghothamachar [a], Michael Dudley [a]

[a] Department of Materials Science & Engineering, Stony Brook University, Stony Brook, NY 11794 USA
[b] Cree Inc., 4600 Silicon Drive, Durham, NC 27703 USA

ARTICLE INFO

Keywords:
A1. Characterization
A1. X-ray topography
A1. Threading mixed dislocations
B2. Silicon carbide

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.

1. 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.

2. 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 100 mm 4H–SiC wafer using monochromatic beam (8.99 keV) with 2° grazing angle. The diffraction vectors are $11\bar{2}8$, $2\bar{1}\bar{1}8$, $1\bar{2}18$, $\bar{1}\bar{1}28$, $\bar{2}118$ and $\bar{1}2\bar{1}8$. The sample-film distance is around 30 cm. Crystals were sent by Cree. Ray tracing simulation

* Corresponding author. Tel.: +1 631 903 0904.
E-mail addresses: jqguo123@gmail.com (J. Guo), yu.yang@stonybrook.edu (Y. Yang), Joe_Sumakeris@cree.com (J. Sumakeris), Robert_Leonard@cree.com (R. Leonard), ouloide.goue@stonybrook.edu (O. Goue), balaji.raghothamachar@stonybrook.edu (B. Raghothamachar), michael.dudley@stonybrook.edu (M. Dudley).

http://dx.doi.org/10.1016/j.jcrysgro.2015.12.028
0022-0248/

was carried out to provide contrast from TMDs with different Burgers vectors on topographs with different diffraction vectors.

3. Results and discussions

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

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

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

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

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. In synchrotron white beam X-ray topography experiment, 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 20 cm 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 $\boldsymbol{g}\cdot\boldsymbol{b}$ 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

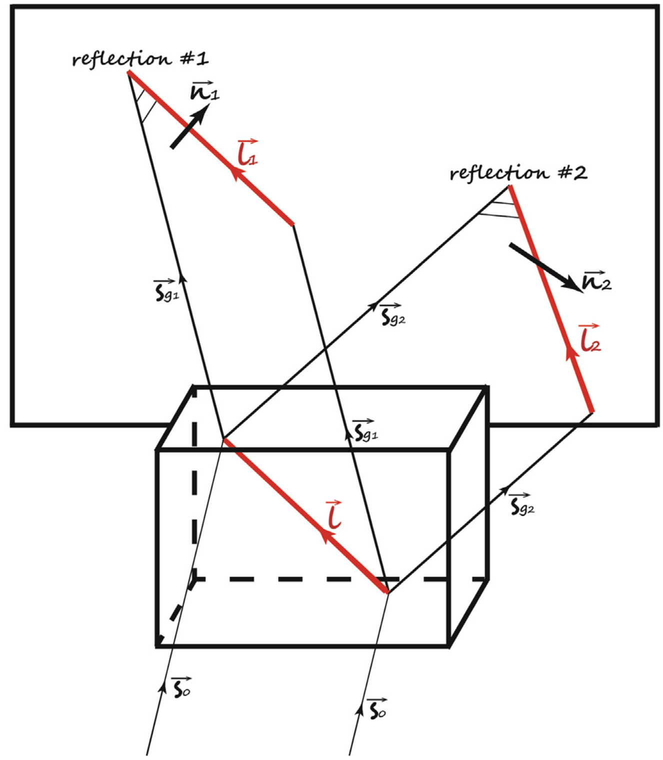

Fig.1. Schematics showing the mechanism of this technique for determination of line directions of dislocations.

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 $\boldsymbol{s}_0$, the diffracted beam directions $\boldsymbol{s}_g$ and the projected line directions $\boldsymbol{l}_1$ and $\boldsymbol{l}_2$ on different reflections can be expressed as vectors with 3-index coordinates. The coordinates for the actual line direction $\boldsymbol{l}$ 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.

3.2. 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

Please cite this article as: J. Guo, et al., Journal of Crystal Growth (2016), http://dx.doi.org/10.1016/j.jcrysgro.2015.12.028