

LACBED Analysis of Threading Dislocation with c+a Burgers Vector in 4H-SiC

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1. Introduction

SiC is an attractive material for power devices owing to its superior properties. However, crystal defects in SiC cause low breakdown voltage, short lifetime and low yield of power devices. The accurate information of defect structure gives us solution how to reduce the defect density. Recently, it is pointed out that threading screw dislocation (TSD) extends to several stacking faults (SFs) [1]. Therefore, clarifying precise structure of TSD strongly required. Several reports suggested the existence of threading mixed (c+a) dislocations, in which the Burgers vector should be represented $\mathbf{b} = n\langle 0001 \rangle + (m/3)\langle 1120 \rangle$ ($n, m = 1, 2, \dots$), in the dislocations currently identified to be the TSD (c-dislocation) as $\mathbf{b} = n\langle 0001 \rangle$ ($n = 1, 2, \dots$) by X-ray topography analysis [2, 3, 4].

However, the existence of threading mixed dislocation is under discussion because this analysis contains ambiguity of Burgers vector and cannot deny the possibility of the dislocation pairs of c-dislocation and a-dislocation (threading edge dislocation: TED) being close to each other. The ambiguity of Burgers vector comes because X-ray topography determines the Burgers vector direction from the image contrast using $\mathbf{g} \cdot \mathbf{b} = 0$ ($\mathbf{g}$: reciprocal lattice vector, $\mathbf{b}$: Burgers vector) but the large anisotropic strain around dislocation in hexagonal SiC disturbs the simple relation $\mathbf{g} \cdot \mathbf{b} = 0$. In addition, the spatial resolution of X-ray topography is not enough to deny the possibility of the dislocation pairs. On the other hand, transmission electron microscopy (TEM) observation using weak-beam dark-field (WBDF) method is suitable for the analysis of the dislocation structures according to its high spatial resolution. Although as mentioned above, it is difficult to accurately determine the Burgers vector direction with the relation $\mathbf{g} \cdot \mathbf{b} = 0$. While, large-angle convergent-beam electron diffraction (LACBED) which is one of the TEM characterization techniques and developed in the dislocation analysis in Si single crystal that can accurately determine the Burgers vector of the dislocation without the influence of large anisotropic strain [5].

In this study, we developed microsampling technique to analyze the targeted threading dislocation (TD), which is identified as a TSD with synchrotron monochromatic beam X-ray topography (SMBXT), for TEM observation. The targeted dislocation was analyzed by using WBDF and LACBED methods and the accurate Burgers vector of the target dislocation was identified.

2. Microsampling technique

Firstly, an X-ray topographic image of 4H-SiC single

crystal is obtained by grazing-incidence SMBXT using 1128 diffraction. SMBXT was carried out at BL08B2 in SPring-8. X-ray energy of monochromatic-beam was 9.53 keV and the angle between the incident beam and substrate surface was ~ 3 degree.

Then, etch pits are formed at the dislocations of the 4H-SiC (0001) surface by KOH etching with Na_2O_2 additive (KN etching) at 510 °C [6] to identify the dislocation position. On the basis of the SMBXT image and an optical microscope image after KN etching, a large hexagonal pit currently interpreted as TSD is chosen as an observation target. At this stage, we can roughly identify dislocation position. However, thickness of cross-sectional TEM specimen ($\sim 200\text{nm}$) is too thin to certainly include the target dislocation in the TEM specimen.

Therefore, focused ion beam (FIB) microsampling technique [7, 8] is applied to preparing the TEM specimen for extracting the area from under the large hexagonal pit. In this process, we select the microsampling position by accurately marking the core of the hexagonal pit. The $15\mu\text{m} \times 15\mu\text{m} \times 200\text{nm}$ plate shape of cross-sectional TEM specimen of the TD was extracted under the etch pit core. The cross-sectional TEM specimen is almost parallel to (1100) plane but slightly tilts [1100] direction for avoiding dislocation missing from the specimen by dislocation tilt in SiC crystal.

The targeted dislocation structure was characterized by LACBED [5] and WBDF methods.

3. Dislocation analysis

Fig. 1 shows the SMBXT image of the 4H-SiC surface taken under the condition of the 1128 diffraction, and large (TSD) and small (TED) white dot contrasts are observed. The encircled large white dot in Fig. 1 is

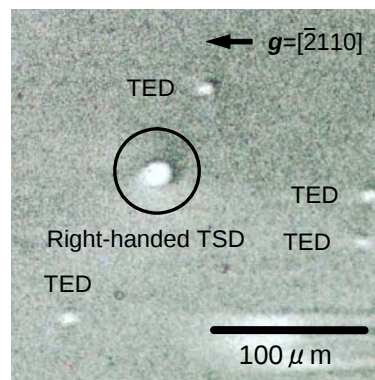


Fig. 1 SMBXT image obtained from a 4H-SiC surface.

determined to be a right-handed TSD with ray tracing simulation [9] by taking account of the reciprocal lattice vector of the incident X-ray.

The cross-sectional TEM specimen of the dislocation under the encircled large white dot in Fig. 1 was fabricated. The dislocation was observed by WBDF method under the $g/3g$ condition using 0001 and 2110 reflections in 4H-SiC. In both 0001 and 2110 reflections, a single line dislocation contrast is clearly observed and no pair of dislocation lines is observed. Thus observed dislocation line is single and not dissociated into the partial dislocation pairs.

Figs. 2 (a)-(c) show the LACBED patterns in various kinds of reciprocal lattice vector obtained from the areas containing the TD. D and H in the figures show the dislocation and the high-order Laue zone (HOLZ) reflection lines, respectively. Because there is the high strain field accompanying the lattice displacement around the dislocation core, the nodes corresponding to the absolute value of the displacement vector will appear with the dislocation line overlapping across the HOLZ line. When the HOLZ line of the reciprocal lattice vector g is overlapped with the dislocation line of the Burgers vector b , measured nodes n satisfies a relation of $g \cdot b = n$ [5]. Thus, the sense and absolute value of the Burgers vector can be determined by measuring the nodes n in 3 kinds of g vector and solving the simultaneous equations from the above

mentioned expression. The HOLZ pattern in Fig. 2 (a) was taken using $11\bar{2} \cdot 8$ reflection. In the intersection of the dislocation line and the HOLZ line, a bright interval between neighboring dark fringes is counted as one node. Observed nodes from the HOLZ patterns using $11\bar{2} \cdot 8$, $11\bar{2} \cdot 8$ and $12\bar{3} \cdot 10$ reflections are 9, -7 and 9 as shown in Fig. 2 (a), (b) and (c), respectively. The absolute value of n indicates the number of nodes and the sign of n show the deviation from the reflection line. In Fig. 2 (a)-(c), the deviations from the reflection lines are same and positive [5]. The Burgers vector ($b=(b_u, b_v, b_t, b_w)$, $b_t=-(b_u+b_v)$) of the TD satisfies all the following equations.

$$-u-v+2t-8w=9 \quad (1)$$

$$-u-v+2t+8w=-7 \quad (2)$$

$$u+2v-3t-10w=9 \quad (3)$$

Burgers vector of the TD was determined to be $b_{uvtw}=[0001]+(1/3)[2110]$ by solving the simultaneous equations. This Burgers vector indicated that the c+a dislocation (i.e. threading mixed dislocation) is included in the dislocations identified as the TSD by SMBXT. The Burgers vector of the c- (screw) component in this threading mixed dislocation is $[0001]$, thus right-handed. Determined rotating direction in c-component by LACBED is consistent with that by SMBXT.

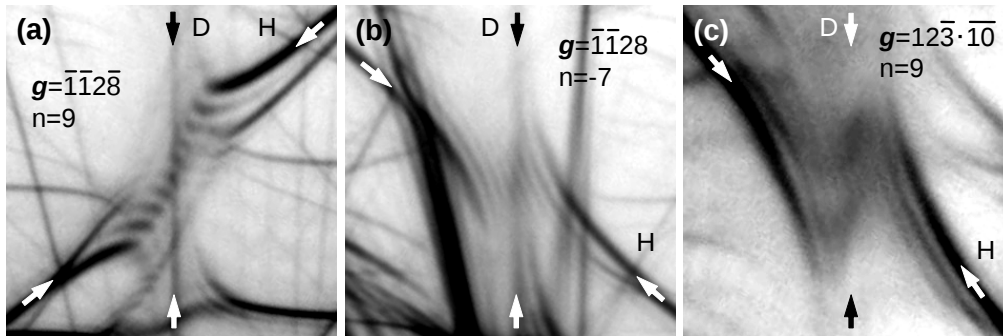


Fig. 2 Bright-field LACBED patterns in several kinds of reciprocal lattice vectors obtained from the region containing the TD. (a) $g=11\bar{2} \cdot 8$, (b) $g=11\bar{2} \cdot 8$, (c) $g=12\bar{3} \cdot 10$.

4. Conclusions

Microsampling technique to observe the targeted dislocation in SiC crystal with TEM was developed. This method is combined SMBXT, KN etching and FIB microsampling technique. The dislocation structure of the TD in 4H-SiC, which was identified as right-handed TSD from the contrast of the SMBXT image, was characterized by LACBED and WBDF methods. It was found that the observed dislocation do not dissociate into the partial dislocation pairs. Furthermore, it was determined that the Burgers vector of the TD is $b=[0001]+(1/3)[2110]$. These results indicate single line threading mixed dislocations with c+a Burgers vector are contained in dislocations identified as a TSD by SMBXT.

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