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Evolution of threading edge dislocation during solution growth of SiC

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1. Introduction

Silicon carbide (SiC) is a promising material for next-generation power device because of its excellent physical properties [1,2]. For the achievement of high-performance SiC power devices, improvement of crystal quality is essential. Defects in SiC are known to degrade the performance of power devices [3-5]. However, commercial SiC crystals, which are grown by physical vapor transport (PVT) method, thousands of dislocations still exist [3]. To achieve high quality SiC crystal growth, we focus on the solution growth method. Solution growth has an advantage to grow high quality crystals because the condition of solution growth is close to thermal equilibrium [6-10]. Recently, we have revealed that almost all threading screw dislocations (TSDs) in seed crystal are converted to Frank-type SFs by step-flow during the solution growth of 4H-SiC. Furthermore, we found threading edge dislocations (TEDs) were also converted to basal plane dislocations (BPDs). However, about half of the TEDs are propagated in the grown crystal. Therefore, in the present study we investigate the TED evolution during the solution growth of 4H-SiC for the complete elimination of the dislocation in SiC.

2. Experimental

4H-SiC was grown in a radio frequency-heated graphite hot-zone furnace (Nisshin-Giken Co., Ltd.) by top-seeded solution growth method. The solution was placed in a graphite crucible and kept in a vertical temperature gradient of 32 K/cm under a high-purity (>99.9999 vol.%) Ar gas flow. The graphite crucible had an inner diameter of 45 mm and was 50 mm high, and the graphite rod was 10 mm in diameter. 4H-SiC(0001) Si face crystals (10 mm × 10 mm) with 4° off-cut towards [$11\bar{2}0$] were used as the seed. The Si for the solvents had a purity of 11N (Tokuyama Co., Ltd.). Carbon was supplied from the graphite crucible. Prior to growth, the 4H–SiC seed crystal and the Si were cleaned by sonication in methanol, acetone, and purified water (18 MΩ·cm). The growth procedure was as follows: (1) the crucible was heated to 1903 K for 1.5 h; (2) the seed crystal, which was mounted on a graphite rod, was immersed in the solution and held there during the growth period; (3) the grown crystal was then removed from the solution. The crucible was rotated by applying the accelerated crucible rotation technique (ACRT). The crucible and the seed crystal were counter rotated with alternating rotation directions. The maximum crucible and seed rotation speeds were 20 rpm in each case. Residual solvent on the crystal was removed by etching in an HNO_3 + HF solution (HNO_3:HF = 2:1).

Surface morphology of the grown crystal was investigated by confocal laser scanning microscope (CLSM) (Olympus LEXT 3100) as well as differential interference contrast (DIC) microscope (Leica DM4000 M) using Nomarski-type prism. Grazing incidence synchrotron reflection X-ray topography was performed using a monochromatic X-ray beam (λ = 0.150 nm) at BL15C in Photon Factory at the High-Energy Accelerator Research Organization, Japan. The applied **g** vector was $11\bar{2}8$. X-ray topography images were captured on a nuclear emulsion plate (Ilford L4, 25 μm)

3. Results and discussion

Figure 1 shows the DIC image obtained from the grown crystal on a vicinal 4H-SiC seed crystal. Macrosteps due to step bunching were observed, which indicates that the growth mode is step-flow growth. CLSM observation revealed that the average macrostep height was 140 nm.

Figure 2 shows the grazing incidence X-ray topography

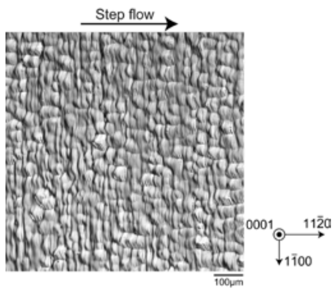

Figure 1. DIC image taken from the grown crystal on a vicinal seed crystal.

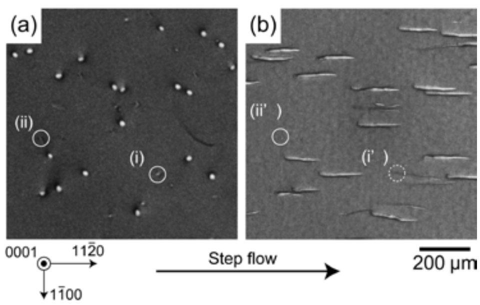

Figure 2. X-ray topography images taken before (a) and after (b) solution growth on the vicinal 4H-SiC seed crystal. TEDs which are imaged as small circular contrast were converted to BPDs imaged as linear contrast as (i) and (i’) for example, while the other TEDs were propagated as (ii) and (ii’) for example.

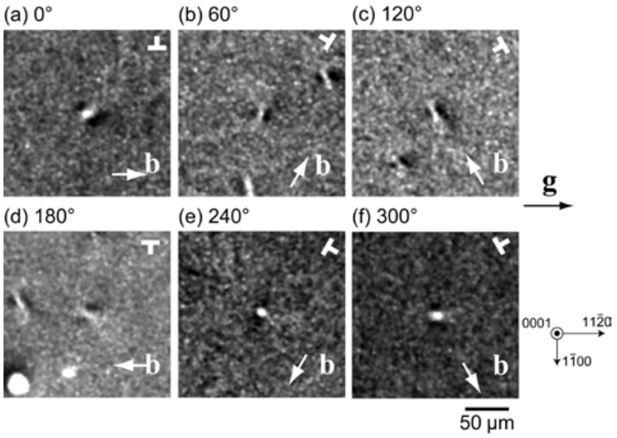

Figure 3. X-ray topography images of TEDs taken before solution growth. (a)-(f) correspond to the six TEDs with different Burgers vectors and angle between the step-flow direction and the Burgers vectors (θ) is 0°, 60°, 120°, 180°, 240° and 300°, respectively.

Table 1. TED/BPD conversion behavior with different angle between the step-flow direction and the Burgers vectors (θ) during solution growth.

TED type	θ (°)	TED/BPD conversion
(a)	0	**Completely**
(b)	60	Never
(c)	120	Never
(d)	180	**Completely**
(e)	240	Partially
(f)	300	Partially

images before and after solution growth. TSDs which are imaged as large circular contrast were converted to Frank-type SFs which are imaged as linear contrast. In addition, some TEDs which are imaged as small circular contrast were converted to BPDs as (i) and (i') for example, while the other TEDs were propagated as (ii) and (ii') for example. To further investigate the TED/BPD conversion behavior during solution growth, we focused on the Burger's vector of TEDs before growth.

In 4H-SiC, the Burgers vector of a TED is 1/3<11$\bar{2}$0>. Therefore, TEDs having six different Burgers vector of 1/3[11$\bar{2}$0], 1/3[$\bar{1}$2$\bar{1}$0], 1/3[$\bar{2}$110], 1/3[$\bar{1}\bar{1}$20], 1/3[1$\bar{2}$10] and 1/3[2$\bar{1}\bar{1}$0] are possible. Although the magnitudes of the Burgers vectors are all the same, the Burgers vector can be identified by high resolution X-ray topography [11]. By comparing the reported X-ray topography images of TEDs in 4H-SiC, we can determine the Burgers vector of TEDs before solution growth as shown in Fig. 3. Angle between the step-flow direction and the Burgers vectors (θ) of type (a), (b), (c), (d), (e) and (f) TED is 0°, 60°, 120°, 180°, 240° and 300°, respectively. Table 1 summarizes the TED/BPD conversion behavior with different Burgers vectors. Type (a) and (d) TEDs were completely converted to BPDs which are pure screw dislocations. Type (e) and (f) TEDs were partly converted to BPD. On the other hand, type (b) and (c) TEDs completely propagated through the grown crystal. Since TEDs whose Burgers vectors are parallel to the step-flow direction are completely converted to BPD, all TEDs can be completely converted to BPD by three different step-flow with the direction of 1/3[11$\bar{2}$0], 1/3[$\bar{1}$2$\bar{1}$0] and 1/3[$\bar{2}$110]. As the growth proceeds, BPDs are excluded to the crystal surface, and finally ultra-high quality SiC without not only TSDs but also TEDs are possibly obtained.

4. Conclusions

We investigated the evolution of TEDs during the solution growth on a vicinal 4H-SiC seed crystal by grazing incidence synchrotron X-ray topography. TEDs with the Burgers vectors parallel to the step-flow direction completely converted to BPDs.

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