Experimental Verification of the Model for Formation of Double Shockley Stacking Faults in Highly Doped Regions of PVT-grown 4H-SiC Wafers

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Abstract

We recently reported on the formation of overlapping rhombus-shaped stacking faults from scratches left over by the chemical mechanical polishing during high temperature annealing of PVT-grown 4H-SiC wafer. These stacking faults are restricted to regions with high N-doped areas of the wafer. The type of these stacking faults were determined

to be Shockley stacking faults by analyzing the behavior of their area contrast using synchrotron white beam x-ray topography studies. A model has been proposed to explain the formation mechanism of the rhombus shaped stacking faults based on double Shockley fault nucleation and propagation. In this paper, we have experimentally verified this model by characterizing the configuration of the bounding partials of the stacking faults on both surfaces using synchrotron topography in back reflection geometry. As predicted by the model, on both the Si and C faces, the leading partials bounding the rhombus-shaped stacking faults are 30° Si-core and the trailing partials are 30° C-core. Using high resolution transmission electron microscopy, we have verified that the enclosed stacking fault is a double Shockley type.

Keywords: B1. 4H-SiC, A1. Double Shockley stacking faults, A1. X-ray topography, A1. Doping, A1. Heat treatment, A1. Rhombus-shaped

Introduction

Silicon carbide (SiC) is well-known as one of the most promising material for next generation power devices. Properties such as high breakdown voltage, wide bandgap, long minority carrier lifetimes [1], high thermal conductivity, high saturated electron velocity and excellent chemical and physical stability [2] make it suitable for applications in high frequency, high power electronic devices [3]. Besides, in optoelectronic applications, SiC offers many competitive advantages as a substrate for III-nitrides from both the epitaxial and the device processing point of view [4]. To realize these predicted performances of SiC devices, high quality of crystal is required. However, variety of crystal defects, such as micropipes and grain boundaries are shown to be detrimental during device operation. Stacking faults, a two dimensional defect generated due to misordered stacking sequence of the basal plane atoms, is also known as a major cause for the device degradation by introducing quantum wells resulting from the local bandgap differences [5]. Bipolar SiC devices with stacking faults suffer from an undesirable forward voltage drop during the device operation, which gradually increased with the time of operation in a constant current. The increase in voltage drop was in the range from several mV to several tenths of a V, in some cases even up to more than 1 V [6]. 4H-SiC material is very susceptible to form stacking faults on basal plane due to the low stacking fault energies, which is about 14.7 kJ m^{-2} [7]. Actually, this tendency mainly owes to the inherent property of polytypism in SiC. Stacking faults in 4H-SiC can be grouped according to different fault vectors: $(a/3)$ type Shockley faults, $(c/2)$ [0001] or $(c/4)$ [0001] type Frank faults, and mixed type faults with both Shockley and Frank fault component fault vectors [8]. Recently, we reported on an observation of overlapping

rhombus- shaped Shockley stacking faults [9]. In this study, we mainly focus on verifying the model developed for explaining the rhombus shaped stacking faults formation by determining the partial dislocation configurations using back reflection synchrotron white beam topography. High resolution transmission electron microscopy of (11-20) cross sectioned specimens has also been utilized to confirm the double layer stacking sequence of these Shockley type faults.

Experiment

The sample is a Nitrogen-doped 4H-SiC wafer which has been subject to annealing ($\sim 1400^{\circ}\text{C}$) after slicing with a 4° off-cut angle along (11-20) direction from a boule grown by PVT method and Chemical Mechanical Polishing (CMP). The wafer is characterized by randomly distributed scratches leftover from polishing and varying doping concentrations across its surface. In previous study, we reported that the rhombus shaped faults bounded with partials are observed only in the high doping regions. The fault vectors are determined to be Shockley type based on $\mathbf{g}\cdot\mathbf{R}$ analysis using synchrotron white beam X-ray topography (SWBXT). Formation mechanism has been proposed as nucleation of dislocation loops and motion of partial dislocations. The model can be verified by determining the nature of the core of each bounding partial dislocation using synchrotron white beam X-ray topography and synchrotron monochromatic beam X-ray topography in back reflection geometry. These experiments were carried out at the topography station at beamline 1-BM, APS and beamline 3.3.2 at ALS. 000 12 reflection images were recorded from the rhombus-shaped partial loops from both Si and C faces of the wafer. In order to reveal the stacking sequence of each fault in atomic resolution, high

resolution transmission electron microscopy (HRTEM) technique has been used. The TEM specimen was cut exactly from the surface scratch on 11-20 cross section plane with a focused ion beam (FIB). The experiments were carried out at Center for Functional Nanomaterials (CFN) of BNL using the experiment tool JEOL2100F.

Results and Discussion

According to our model [9], the rhombus shaped stacking faults result from the expansion of small hexagonal dislocation loops nucleated from surface scratches below the brittle-ductile-transition temperature for 4H-SiC, as illustrated in Fig. 1. All the faults are determined to be Shockley type and the burgers vector for their bounding partial dislocations are determined to be $1/3\langle 1-100 \rangle$ based on **g.R** and **g.b** contrast analysis accordingly [9]. Under the driving force of thermal energy, and the different mobilities of the Si and C core dislocations, as well as that of 30° and 90° character dislocation segments, the bounding partial of the faults ends up with a rhombus shape, with two 30° Si-core as leading and two 30° C-core as trailing partials. Papers report by Kuhr [10] and observations by Pirouz [11] argue that when 4H-SiC is highly doped, the presence of stacking faults can lower the energy of electron state in the faulted crystal compared to the perfect crystal. Double fault has even lower energy level than single fault. Calculations showed that a faulted crystal containing these double Shockley faults is more stable than the perfect 4H-SiC crystal at temperatures greater than 1000°C provided a threshold doping level of around $2 \times 10^{19}\text{cm}^{-3}$ is exceeded. We observed such stacking fault formation in more highly doped regions suggesting that the doping level in those regions exceeds this threshold value. If the barrier to the motion of the Shockley

partial dislocations in this doping level can be overcome, such as by a high temperature treatment in our case, such highly doped crystals are expected to spontaneously fault via the expansion of Double Shockley faults [9].

Therefore, the proposed model can be validated by determining the core structures of each bounding partial dislocation comprising the rhombus partial dislocation loop. Using back reflection X-ray topography, the signs of partial dislocations can be identified by the distinct contrast features generated by the opposite signs of the edge Burgers vector component. Therefore, in back reflection, by comparing the observed image with the result predicted by the ray tracing method [12], the partial core structure can be determined. Fig. 2 is a schematic of the geometry of recording and the resultant simulated images. The concave diffracting plane due to the extra half plane of dislocation extending away from the surface tends to focus the diffracted x rays into a narrow black line on the detector, while the convex diffracting plane associated with an extra half plane extending towards the surface give rise to a white stripe with the defocused x rays overlapping at the edges of the stripe.

Fig. 3 is a [11-20] projection illustrating the atomic arrangements of the Si and C core partial dislocations inside the 4H-SiC crystal. If an extra half plane associated with a partial terminated on Si core, it is a Si-core partial and this extra half plane should extend toward Si face. Thus Si partial will exhibit white contrast when the image is recorded from the Si face but black contrast from C face. Conversely, C-core partial with extra half plane towards C face will exhibit black contrast while recording from Si face but white contrast from C face. X-ray topography experimental observations are shown in Fig. 4.

Fig 4. (a) and (b) are 11-20 and 1-100 transmission images provides an overall reference

for comparison. The down step direction is pointing down inside the picture. Fig. 4 (c) and (e) are the 000 12 back reflection images recorded from the corresponding areas inside the blue and the yellow squares in Fig. 4(a) and (b) respectively. In Fig. 4(c), loops expanding toward upstep are determined to be generated from C face, while in Fig. 4(e) loops expanding toward downstep should be generated from Si face. Regardless of which face the partial loops were generated from, the leading partials are always of Si-core nature while the trailing sessile C-core segments are left in their wake. In Fig. 4(c) recorded from C face, therefore as predicted, the trailing partials exhibit white contrast confirming their C core nature, and the leading partials exhibit dark contrast confirming their Si core nature. Conversely, in Fig. 4(e) recorded from Si face, the trailing partials exhibiting dark contrast are C core, whereas the leading ones with white contrast are Si core. Fig. 4(d) and (f) are the schematics corresponding to the experimental images in Fig. 4(c) and (e) above. Due to the penetration depth in this geometry is 12.5um which delineated as dashed lines marked inside the picture, the contrast from partials below the dashed white line in Fig. 4(d) and above the dashed line in Fig. 4(f) is not visible in the recorded image Fig. 4(c) and (e).

HRTEM micrographs recorded from the stacking faults show a (62) stacking sequence confirming the presence of double Shockley faults formed by the glide of leading Shockley partials on adjacent basal planes. Driven by thermal energy, the glide of the leading partial leads to a local transformation from 4H stacking sequence (ABA'C'ABA'C'...) to 3C stacking sequence (ABCABC....). The stacking sequence of the faults is effectively six layers of 3C stacking sequence.

Conclusion

By adopting the back reflection recording geometry of synchrotron X-ray topography, we have determined the core structure of the Shockley partial dislocations bounding rhombus shaped Shockley stacking faults that are generated from surface scratches in highly doped regions during heat treatment. The core structure has been verified to be consistent with the model for formation of the rhombus shaped faults from expansion of hexagonal dislocation loops developed in our previous study. Using HRTEM, the faults are also verified to be double layered Shockley faults formed by the gliding of Shockley partials in two adjacent planes.

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Figure(s)

Figure 1. (a) initial hexagonal partial dislocation loop (b) rhombus shape partial dislocation loop comprising of 30° Si and C cores left over by the 90° Si core partial moving out from the initial hexagonal partial dislocation loop (a)

Figure 2. In back reflection, the partial dislocation exhibits black contrast (i.e. the core causes convergence in the locally diffracted rays) if it extends away from that surface (a) , and white contrast (i.e. the core causes divergence in the locally diffracted rays) if the extra half-plane extends toward the imaged surface (b) .

Figure 3. A schematic [11-20] projection showing the arrangement of Si and C core partial dislocations with respect to the Si and C faces inside the 4H-SiC crystal

Figure 4. (a) and (b) are (11-20) and (1-100) reflection images recorded in transmission geometry from the same region showing for comparison. (c) and (e) are the 000 12 back reflection images recorded from the corresponding areas inside the blue and the yellow squares in (a) and (b) respectively. (c) recorded from C face, the partials exhibit white contrast are C core, the ones exhibit dark contrast are Si core. Conversely, in (e) recorded from Si face, the partials exhibit dark contrast are C core, whereas the ones with white contrast are Si core. (d) and (f) are the schematics correspond to their experimental images (c) and (e) above. Due to the limitation of the penetration depth in this geometry, the contrast from partials below the

dashed white line in (d) and above the dashed line in (f) is not visible in the recorded image (c) and (d).

Figure 5. HRTEM image showing the stacking sequence of predicted six layer 3C structure formed by gliding of double Shockley partials