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Atomic configuration of irradiation-induced planar defects in 3C-SiC

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The atomic configuration of irradiation-induced planar defects in single crystal 3C-SiC at high irradiation temperatures was shown in this research. A spherical aberration corrected scanning transmission electron microscope provided images of individual silicon and carbon atoms by the annular bright-field (ABF) method. Two types of irradiation-induced planar defects were observed in the ABF images including the extrinsic stacking fault loop with two offset Si-C bilayers and the intrinsic stacking fault loop with one offset Si-C bilayer. The results are in good agreement with images simulated under identical conditions. © 2014 AIP Publishing LLC.

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Cubic silicon carbide (3C-SiC) is a promising material for nuclear applications due to its excellent mechanical properties and good corrosion resistance at very high irradiation temperatures.1,2 In previous studies, the evolution of defects induced by neutron and Si-ion irradiation under different irradiation temperatures was found to be similar.3 When SiC is irradiated at temperatures above 1000 °C, small defects tend to aggregate to become planar defects, which are identified as Frank loops by transmission electron microscopy (TEM).4–7 The size of these loops increases, and the number density decreases with elevated temperatures. Recently, it has been found that irradiation-induced planar defects include not only the interstitial loop but also the vacancy loop in Si-ion-irradiated single crystal 3C-SiC.8 Although atomic models of these irradiation-induced planar defects have been predicted, there have not yet been any direct evidences which confirm the atomic configuration of these faulted loops.

The material used for this research was a single crystal 3C-SiC grown on a Si substrate via chemical vapor deposition (NOVASiC, France). The specimens were irradiated by 7 MeV Si13+ ions using a 9SDH-2 Tandem Accelerator, which was designed and developed by American NEC,9 at National Tsing-Hua University. The irradiation conditions were set with a fluence of $2 \times 10^{17}$ ion/cm² each at irradiation temperatures of 1000, 1200, and 1350 °C under vacuum (10⁻⁶ Torr), with the rate of displacement per atom at about 6 × 10⁻⁵ dpa/s at the depth regime of 20 dpa. Irradiation temperatures were measured on the irradiated surface by infrared pyrometer and confirmed by thermocouple. Furthermore, The profile of implanted Silicon ion was simulated with SRIM program, and the displacement energies of Si and C were 35 eV and 21 eV, respectively.10 We have characterized the structure of irradiation-induced defects using annular bright-field (ABF) imaging technique performed in a spherical aberration corrected scanning TEM (Cs-corrected STEM). The STEM ABF is able to directly detect atom position of light atoms, e.g., oxygen, lithium, and carbon, which cannot be imaged by high-angle annular dark field (HAADF) images.11–13 Experiments were performed with a Cs-corrected STEM (JEOL, JEM-ARM200F) at an accelerating voltage of 200 kV. Thin foil specimens for TEM analysis were prepared for cross-sectional observation by mechanical polishing followed by the 3–5 keV Ar ion-milling method. Structural models of the atomic arrangement were built by CrystalKitX software, and STEM image simulation was carried out with the help of MacTempasX software.

Irradiation-induced planar defects on the {111} plane were observed in all of the three samples at irradiation temperatures of 1000, 1200, and 1350 °C. Discrepancies in the size and number density of defects between the different samples were noted (Table I). Two types of loops, both with layer displacement, were observed in the ABF images (Figs. 1 and 2). The layer sequence of the two types of loops conforms to the most common stacking faults in fcc materials, which are intrinsic and extrinsic stacking faults (ISF and ESF).14 The finding of streaks in the inset of a fast Fourier transform (FFT) pattern transformed from Figs. 1(a) and 2(a), indicates the existence of stacking faults in the atomic resolution image. Large stacking faults (>300 nm) formed in the 3C-SiC layers grown by CVD on silicon are mainly due to the large lattice mismatch and the difference in the thermal expansion coefficients between Si and SiC.15,16 Although stacking faults were also found inside the irradiation-induced loops, the radius of the loops are much smaller (<20 nm) than the stacking faults formed during crystal growth. Furthermore, the formation of stacking faults inside irradiation-induced loops is because of the condensation of interstitial or vacancy atoms during high temperature irradiation. Owing to the difference between the stacking faults formed during irradiation and crystal growth, the two types of irradiation-induced loops observed in our study are...
called extrinsic stacking fault loops and intrinsic stacking fault loops, respectively.

Stacking faults in a 3C-SiC crystal occur when the stacking sequence differs from the cubic closed-pack stacking arrangement (ABCABC…). It is worth noting that any one layer in this stacking sequence may have a local cubic or hexagonal environment. In order to indicate the hexagonal environment in which the tetrahedron unit structure (SiC₄ or CSi₄) in SiC is rotated by 180°/C₁₄ along the {111} plane, a prime was superposed to the notation, i.e., the tetrahedron unit structures on the A₀ site is rotated by 180°/C₁₄ from A site.

In Fig. 3, the magnified image of an intrinsic stacking faulted loop shows the faulted planes with one offset Si-C bilayer. The spots with darker contrasts indicate Si atoms, while carbon atoms appear as lighter contrasts. Our STEM ABF images were taken with a convergence angle about 25 mrad and collection semi-angle in the range of 11–23 mrad. Images were post-processed by HREM filter in Gatan Digital Micrograph software to de-noise and improve images. An intrinsic stacking fault may be developed in a 3C-SiC crystal by removing part of a close-packed plane and producing the stacking sequence ABCjjBCABC… Such a method of developing an intrinsic stacking fault is physically possible and can occur by the condensation of vacancies on the {111} planes. Furthermore, if a layer was removed or added in the ideal stacking sequence, a short range tensile or compressive strain might be induced. To relieve these strains, parallel planes along the stacking sequence near the offset interface have tendency to glide along {111} planes. The movement of layers across the slip plane of a 3C-SiC crystal will then produce the stacking sequence ABCB₀CABC…, for an intrinsic stacking fault loop.

Another planar defect, an extrinsic stacking faulted loop, was also characterized by STEM ABF imaging. Fig. 4 shows an extrinsic stacking faulted loop with two offset Si-C bilayers. The imaging conditions were the same as for the ABF image of the intrinsic stacking fault loop, and filtering by a HREM filter was also carried out. This result suggests that once irradiation-induced interstitials agglomerate on the {111} plane to form an added layer, the stacking sequence will change from ABCABC… to ABC/B/ABC… Owing to the strain caused by the additional plane, other {111} planes near the added layer or the plane itself tends to glide along {111} planes to relieve this strain. The stacking

| Table I. Quantification of the two types of planar defects. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
|                | Loop diameter (nm) | Loop density (#/m³) |
| T (°C) | Dose | ESFᵃ | ISFᵇ | ESF | ISF |
| 1000   | 20 dpa | 4.7  | 2.6  | 1.6 x 10²³ | 5.3 x 10²² |
| 1200   | 20 dpa | 9.8  | 4.5  | 4.8 x 10²² | 2.1 x 10²² |
| 1350   | 20 dpa | 18.9 | 6.5  | 2.7 x 10²² | 0.9 x 10²² |

ᵃESF: Extrinsic stacking fault loop.
ᵇISF: Intrinsic stacking fault loop.
sequence then becomes ABCB’ A’BC…. In terms of polytypic transformations in SiC, stacking sequences were found in ISF and ESF loops, respectively. It is well understood that the occurrence and stability of 2H and 4H are higher than 3C over the temperature range of 1000 °C to 1400 °C. Moreover, very low or negative ISF and ESF energy obtained by molecular dynamics and ab initio simulation also supports this observation.14,24

Images simulated under identical conditions were in agreement with the experimental images. In Figs. 5(b) and 5(e), simulated ESF and ISF ABF images are shown for inner and outer apertures at 11 and 23 mrad, respectively. The best results are obtained at zero defocus and a 25.5 mrad probe semi-angle for both simulations corresponding to a probe size of 0.15 nm. We speculated that the lower contrast shown in both offset layers in the ESF and ISF ABF experimental images may be because of the overlap of Si/C atoms on the primitive site and displaced atoms which have undergone a hexagonal operation. The stacking fault loops should be disk-shaped with a radius of less than 15 nm and lie in [111] planes surrounded by an un-faulted structure. Whereas our TEM sample is thicker than 15 nm, an overlap of atoms may occur and impart the contrast.

In summary, we used a spherical aberration corrected (Cs-corrected) STEM with ABF imaging technique to characterize the irradiation-induced planar defects. These defects are extrinsic stacking fault loops with two offset Si-C bilayers and intrinsic stacking fault loops with one offset Si-C bilayer. The formation of stacking fault loops is in good agreement with the very low or negative simulated stacking fault energy in 3C-SiC and the stability of different SiC polytypes. Overall, our observation on the microstructural configuration has provided a better understanding of planar defect structures and the evolution of radiation damage in 3C-SiC.

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