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High-Resolution Scanning Transmission Electron Microscopy Study of Black Spot Defects in Ion Irradiated Silicon Carbide

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Silicon carbide is of great interest as a structural material in nuclear systems because of its high strength, corrosion resistance and high thermal conductivity. An important material property related to failure is irradiation induced volume increase, or swelling. Swelling saturates in silicon carbide at relatively low irradiation temperatures (~< 1000 °C) and doses (~< 10 dpa or displacements per atom), when the major defects appear in bright field transmission electron microscopy (TEM) images as nanometer scale black spots and are referred to as black spot defects (BSD) [1]. The detailed internal structure of BSD is unknown. We are working towards understanding the structures and evolution of BSD by combining high resolution scanning transmission electron microscopy (STEM) and defect structure modeling [2].

We have irradiated single-crystal 4H-SiC and polycrystalline 3C-SiC with 3.15 MeV carbon ions to a dose of 5.14×10^{16} C/cm^2 at 600 °C, 800 °C, and 950 °C. The corresponding average damage level is 0.5 dpa. TEM observation was performed at 300 kV in a FEI Tecnai TF30 and STEM observation was performed at 200 kV in a probe Cs-corrected FEI Titan.

Figure 1a shows the typical bright field TEM image of BSD in 4H-SiC irradiated with carbon at 950 °C. The spotty contrasts are black spots defects. From the [1120] zone, multi-beam bright field images, the average size of the BSD in the carbon-irradiated 4H-SiC samples is determined to be 1.3 ± 0.8 nm, 1.5 ± 1.1 nm, 1.4 ± 1.1 nm for irradiations performed at temperatures of 600°C, 800 °C, and 950 °C, respectively. Figure 1b shows the number density of BSD in carbon-irradiated 4H-SiC samples as a function of the irradiation temperature.

Figure 2 shows typical STEM images of the irradiated SiC samples. Figure 2a, a high angle annular dark field (HAADF) image, which emphasizes Z-contrast, with the 24.5 mrad semi-convergence angle electron probe and the 54 – 270 mrad collection angle, shows periodic silicon columns in a 4H-SiC irradiated with carbon at 600 °C. Figure 2b is an annular dark field (ADF) image, emphasizing strain contrast, of 17.5 mrad semi-convergence angle and 29 – 144 mrad collection angle from the same sample. It shows dark spotty contrasts from BSD. This result suggests that BSD are clusters of low-Z point defects, possibly C interstitials. Figure 2c-e show the simulated structure of a tri-carbon interstitial defect [2] and its STEM image in a very thin simulated specimen. The models show that the brightness of Si columns near the carbon interstitials is lower than that in the matrix, and is consistent with the experimentally observed lower intensity of BSD.

The size of BSDs does not change significantly for irradiations performed in the range of 600 °C to 950 °C. However, the density of BSD decreases with irradiation temperature, suggesting that annealing annihilates these defects. There is no major growth or coalition of defect clusters from 600 °C to 950 °C.

Ongoing HRSTEM study on the size, shape, and strain of BSD will provide valuable information about the structure of BSD.

References:

[1] Lance L. Snead *et al,* Journal of Nuclear Materials **371** (2007), p. 329.
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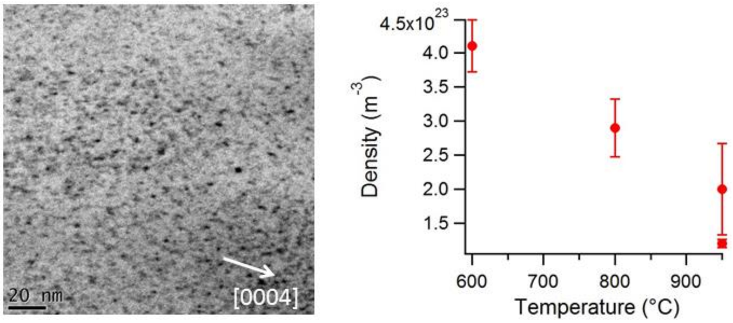
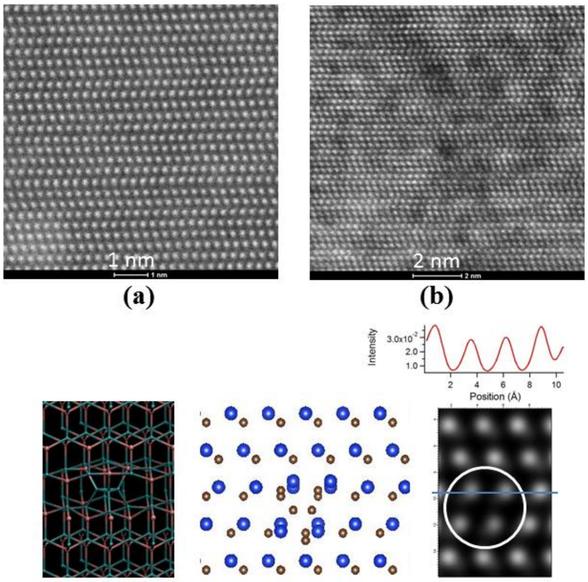


(a) **(b)**

Figure 1. (a) [1120] zone [0004] two-beam condition bright field TEM image of 4H-SiC irradiated with carbon at 950 °C. The irradiation level at the image region is about 0.4 dpa. (b) Black spot defect density in carbon irradiated 4H-SiC at different temperatures, all at 0.4 dpa.



(a) **(b)**

(c) **(d)** **(e)**

Figure 2. Experimental STEM images (a-b) and image simulation from a tri-carbon interstitial model (c-e). (a) HAADF images of a 4H-SiC sample irradiated with 3.15 MeV carbon ions at 600 °C. See text for the imaging conditions. (b) ADF image of the same sample. (c) Model of a three-carbon interstitial cluster in 4H-SiC [2]. (d) Projection of the defect model along the [1120] axis. (e) Simulated STEM image from the defect model, with the same imaging condition as in (b). The sample thickness is 3 nm. The interstitial cluster location is circled, and the graph is an intensity profile along the blue line.