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SILICON CARBIDE AMORPHIZATION BY ELECTRON IRRADIATION

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Observations made more than ten years ago¹ showed that SiC could be made amorphous at cryogenic temperatures by in-situ 300kV electron irradiation. However, high-voltage electron microscope (HVEM) results² indicate a threshold voltage of 725 kV for amorphization of SiC at 140 K. In addition, a recent review³ exposes the considerable uncertainty in the literature regarding displacement energies for SiC. Therefore, further experiments have been performed in a Philips CM30 (LaB₆ cathode) with a Gatan double-tilt cooling holder in an attempt to determine the threshold voltage for amorphization at ~140 K. Sintered α -SiC (defected 6H polytype), beam direction $B = \langle 11\bar{2}0 \rangle$, and probes containing ~75 nA in ~0.5 μm , were used. Amorphization occurred in <10 min at 300 kV and after ~60 min at 180 kV (Fig. 1); visible darkening occurred at lower voltages and doses. Similar behavior occurred for $B = [0001]$. The critical dose for amorphization was measured as a function of accelerating voltage. Probe current profiles were measured by post-specimen scanning (CM30 SCIM mode with 100 μm -diameter Gatan STEM detector) images of the focussed probes positioned in a hole, and probe currents were measured from the exposure time, which had previously been calibrated with a Faraday cup. The current density i at radius r in a Gaussian probe of full-width-half-maximum (FWHM) D and total current A is:

$$i = [4A \ln 2 / \pi D^2] \exp [-4 \ln 2 (r / D)^2] \quad (1)$$

Measurement of the radius judged to be fully amorphous at a measured irradiation time thus allows the critical dose for amorphization to be calculated. Values are shown in Fig. 1. A quantitative reconciliation of these data with the HVEM results of Inui et al.² through a model that explicitly considers different displacement thresholds for different atomic species⁴ was not possible because the shape of the HVEM data is inconsistent with calculated displacement cross-sections. However, if the apparent 180 kV threshold is associated with carbon displacements, the carbon displacement energy E_d is <37 eV, in fair agreement with experimental estimates³ and recent molecular dynamics calculations.⁵

Near the threshold voltage, because the displacement rate decreases sharply, the critical dose for amorphization can require excessively long irradiation times. In an attempt to overcome this limitation and refine the threshold voltage, experiments were performed in a Philips CM200FEG at much higher current densities (and thinner regions). Amorphization was achieved in only a few seconds with the most intense probes (Fig. 2), but amorphization occurred at 160 kV and even 120 kV with critical doses that were a small fraction of those shown in Fig. 1. Amorphization was also found to proceed more rapidly in thinner regions of the specimen (Fig. 3). These results indicate that surface effects and possibly high dose-rates are creating dominant artifacts. Although thin surface oxide layers may play a role, sputtering at the exit surface (the probable main cause of hole-drilling in metals such as steel at 200kV) may lead to a local composition change and nucleation of amorphous material which then grows in the beam direction through easier displacements at the amorphous-crystalline interface. These considerations prompted time-series PEELS measurements of composition during amorphization (EMiSPEC Vision, Gatan PEELS, CM200FEG) and measurements of t/λ by energy-filtered imaging (CM30, Gatan Imaging Filter), but no conclusive evidence of sputtering effects was obtained.

In-situ annealing experiments were performed with a Gatan single-tilt heating holder in a Philips CM200FEG to observe the crystallization of amorphized regions. No changes were observed for temperatures up to ~1200 K. At 1240 K the amorphous zones produced at 300 kV (~250 nm dia.) and 200 kV (~100 nm dia.) crystallized into highly defected epitactic SiC by the movement of the amorphous-crystalline interface at ~5 nm/min (see Fig. 4). However, crystallization proceeded more slowly along [0001]. In larger amorphized regions (>500 nm) small amounts of non-epitactic nucleation and growth were also observed. These annealing results are in general agreement with those of Inui et al.^{2,6} but are quite different from the in-situ annealing behavior of cross-sectioned specimens amorphized by ion implantation with Cr⁷ and Fe.^{8,9}

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FIG. 1 - Accelerating voltage dependence of critical amorphization dose for SiC at $\langle 1120 \rangle$.
 FIG. 2 - SiC amorphized at a) 200 kV, 7.3 nA b) 160 kV, 16 nA. Critical dose $\sim 1 \times 10^{27}$ e/m².
 FIG. 3 - Thickness dependence of amorphization rate at 200 kV, ~ 30 nA in 100 nm probe, 120 s.
 FIG. 4 - In-situ annealing of SiC amorphized at 200 kV.

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