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In situ Transmission Electron Microscopy of Room-temperature Plastic Deformation and Recovery in Thin 3C-SiC

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Silicon carbide (SiC) is an excellent candidate material for microelectromechanical systems (MEMS) and nanoelectromechanical systems (NEMS) due to its outstanding electrical, physical, and chemical properties under harsh working environments [1]. It also has high irradiation resistance, good thermal conductivity, and chemical inertness, which make SiC a promising material for next generation nuclear reactors, such as the cladding of tri-structural isotropic (TRISO) particles for very high-temperature reactors [2][3]. Understanding the mechanical properties of SiC under stress is important to predict its performance in those applications. Here, we report *in situ* nanoindentation tests on 3C-SiC, which has cubic crystal structure, in a transmission electron microscopy (TEM) to understand the mechanisms behind its small length scale mechanical properties.

3C-SiC bulk samples were made by chemical vapor deposition and were polished before TEM sample preparation. Standard TEM samples were fabricated using focused ion beam (FIB, Zeiss Auriga) with 30 keV / 20 pA as the last polishing step to a final sample thickness of around 100 nm. Standard samples were observed in TEM (Tecnai TF-30, 300 keV) to characterize the as-synthesized microstructure of the material. Samples for *in situ* indentation tests started with pieces about 1 μm thickness (15 μm x 7 μm), lifted out from the bulk using an Omniprobe micromanipulator in the FIB. The pieces were welded on Si wedge-shaped substrates (Hysitron silicon wedge 1 μm plateau) and thinned down to 150~200 nm with 5 keV / 100 pA as the last polishing current. *In situ* indentation tests were performed in TEM with single tilt Picoindenter holder (Hysitron PI 95 Picoindenter) operated in displacement control mode with velocity ~1 nm/s. Force-displacement curves and videos of the samples were obtained simultaneously.

In a typical test without microstructure near the indent, a rosette pattern of dislocations formed ahead of the indenter tip, as shown in Figure 1. Figure 2 shows a series of still images from an *in situ* nanoindentation test with a grain boundary, in which the rosette pattern is visible on the far side of the boundary (Figure 2d-f). In the video, the dislocations in the rosette pattern have the same shape and move in the same direction, indicating that they share the same Burgers vector. The corresponding load-displacement curve shown in Figure 2a shows room-temperature plasticity during loading, which is not common for ceramics. This result suggests that the brittle to ductile transition temperature of free-standing thin 3C-SiC (the TEM sample) under indentation is below room temperature. Reduced transition temperature at small length scales has been observed in 6H-SiC [4] and 4H-SiC [5]. Plasticity is derived from dislocation generation and movement, since the samples yielded in the load-displacement curves when newly-created dislocations started to move in the TEM images.

Indentation on samples containing grain boundaries, as in Figure 2, shows higher yield strength compared to samples without grain boundaries near the indent as in Figure 1. TEM images show that grain boundaries pin the dislocations. Figure 2c shows the moment just before de-pinning, and Figure 2d shows the subsequent moment. After crossing the boundary, the perfect dislocations dissociated, creating the dark ribbons shown in Figure 2e. We believe that the dislocations dissociated into partial dislocations with stacking faults past the grain boundary because they were no longer subject to the grain boundary force, which had balanced the repulsive force of the two potential partial dislocations.

On unloading, the plastic deformation recovered, as the newly-generated dislocations went back to the surface where they had been generated and almost all of them ultimately annihilated, as shown in Figure 2f-g. A possible mechanism for plastic deformation recovery is that bowing of the dislocations under loading, created by pinning at the surface, creates line tension that tends to straighten the dislocations during unloading, driving them back to their nucleation point [6].

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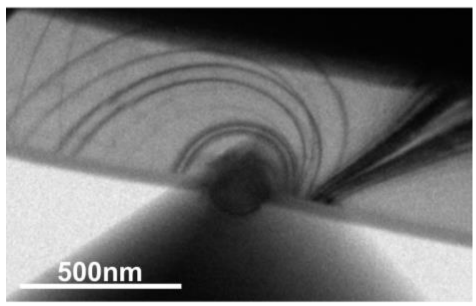

Figure 1. Bright-field TEM image of rosette pattern of dislocations generated by the indenter.

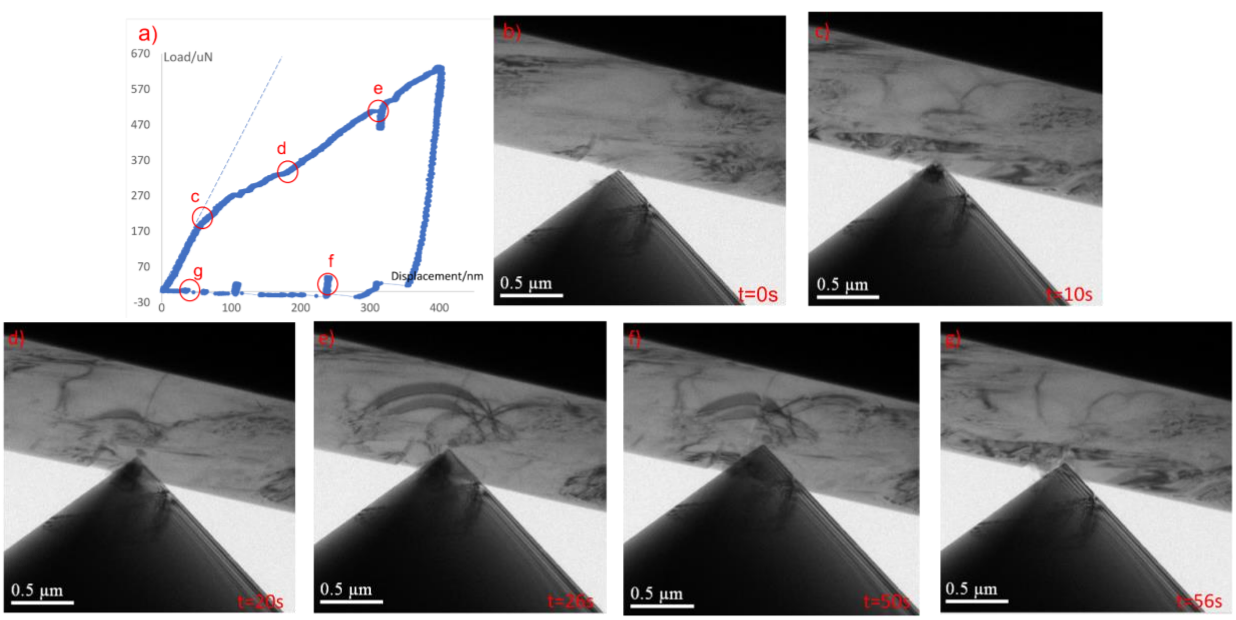

Figure 2. Load-displacement curve and corresponding bright-field TEM image sequences of *in situ* indentation test on 3C-SiC with a grain boundary

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