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A Multi-Step Transmission Electron Microscopy Sample Preparation Technique for Indented Ceramics having Extensive Sub-Surface Cracking

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Understanding the deformation mechanisms in ceramic materials is crucial for optimizing and implementing next-generation ceramic materials in body and vehicle armor systems. One particularly powerful tool for studying deformation mechanisms is transmission electron microscopy (TEM) of impacted materials. Indentation is useful for studying deformation mechanisms, since this method offers the ability to track microstructural features as a function of depth from the indent. Preparing an indented sample for examination in TEM is extremely challenging since the sample preparation process may introduce additional cracks and artifacts. Ion beam milling processes can also induce surface damage and artifacts, such as amorphization and re-deposition of amorphous material in and on indented ceramics [1]. Amorphization should particularly be minimized, since amorphization is an inelastic deformation mechanism that has been observed in some ceramics [2]. All of these effects will change the appearance of the indentation-induced cracking and other microstructural features, therefore obscuring the "true" microstructure of the mechanically damaged area. The goal of this work was to develop a sample preparation technique for heavily cracked ceramics that preserves the true microstructure for TEM imaging and analysis.

In this study, we indented silicon carbide (SiC) with several Knoop indentations using a Tukon 2100B hardness tester. SiC samples were cut and polished, and then indented with a series of 2.9N load indentations, spaced 100 μm apart. The sample was transferred to an FEI Nova600 dual beam focused ion beam (FIB) instrument where it was ion milled using either the H-bar geometry or the lift-out method. During initial ion milling of the indented SiC, the sample developed expansive milling-induced cracks, which often resulted in sample breakage. Milling-induced cracking should be avoided, since it would be difficult to distinguish cracks due to the indentation from cracks from the preparation process. Therefore, we used a low-viscosity epoxy to fill in the cracked areas and hold the sample together during the sample preparation process. The epoxy has an additional advantage in that it reduced the amount of redeposited amorphous material into the cracks and voids.

To infiltrate the epoxy into the cracks, the sample was first tripod polished to ~50μm and the plane of the polish adjusted so that it was parallel to the line of indents (Figure 1a). This initial polishing step is necessary because the cracks under the indents did not emanate from the top surface, so the epoxy must be infiltrated from the side (cross-sectional) surface. In order to fully infiltrate the epoxy into the network of sub-surface cracks, we first revealed the cracked regions using the FIB, as shown schematically in Figure 1b, with the opened cracks shown in Figure 2a. Without this step the cracks can become filled in and obscured during mechanical polishing. After ion milling to open the cracks, a Buehler Cast 'N Vac 1000 Vacuum Impregnation System was used to vacuum infiltrate a low viscosity epoxy into the cracks. The sample was suspended in an inverted position during the epoxy infiltration (Figure 1c). The indented SiC/epoxy infiltrated sample was then thinned to electron transparency using the FIB in the second ion milling step, as shown in Figure 1d. Figure 2 shows SEM images of the cracks under the indent before (a) and after (b) epoxy infiltration. In Figure 2c, the corresponding TEM image

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is shown, demonstrating that the cracks are completely filled with epoxy. Figure 3 shows two representative TEM images, with a high density of stacking faults (3a) and dislocations (3b). These images demonstrate that our sample preparation method is suitable for the identification of deformation mechanisms. We have developed a new TEM sample preparation procedure for indented ceramics. SiC TEM samples prepared using this method can be made very thin (~145 nm), and the defects (such as stacking faults and dislocations) can be observed and mapped by their distance from the indent.

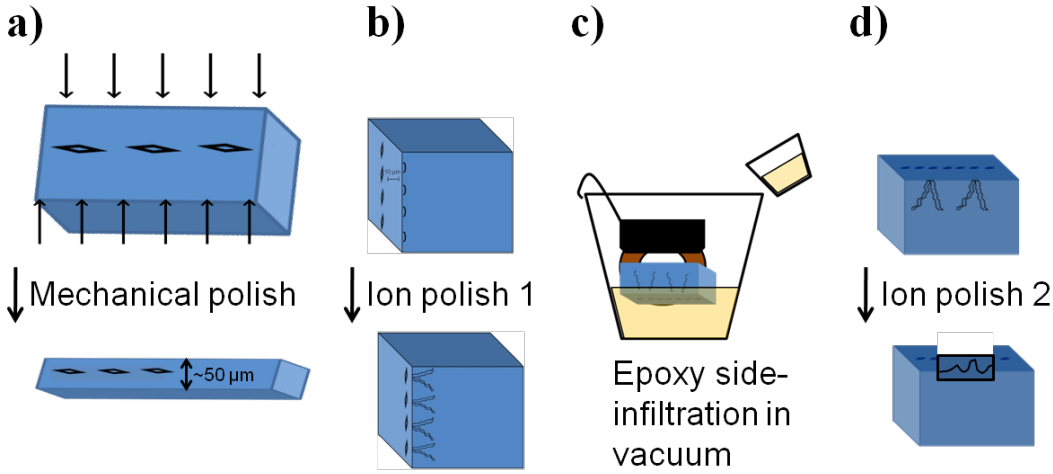

Figure 1. A schematic of the sample prep process, including mechanical polishing (a), ion milling to open up the cracks (b), epoxy infiltration (c), and final ion polishing (d).

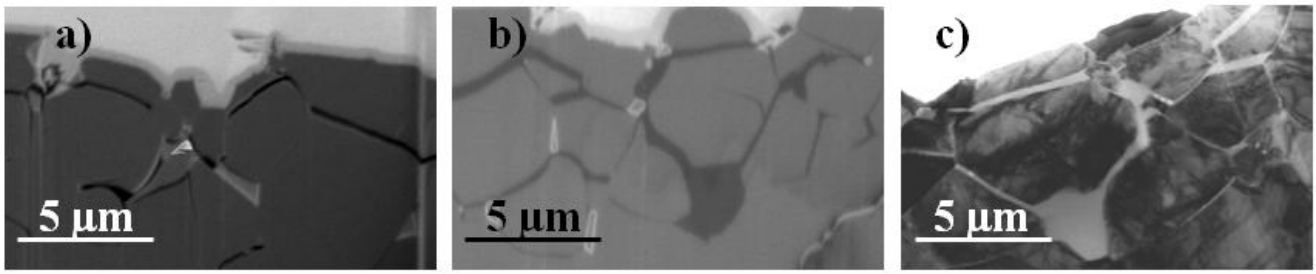

Figure 2. SEM images before (a) and after (b) epoxy infiltration, and the resultant TEM image (c).

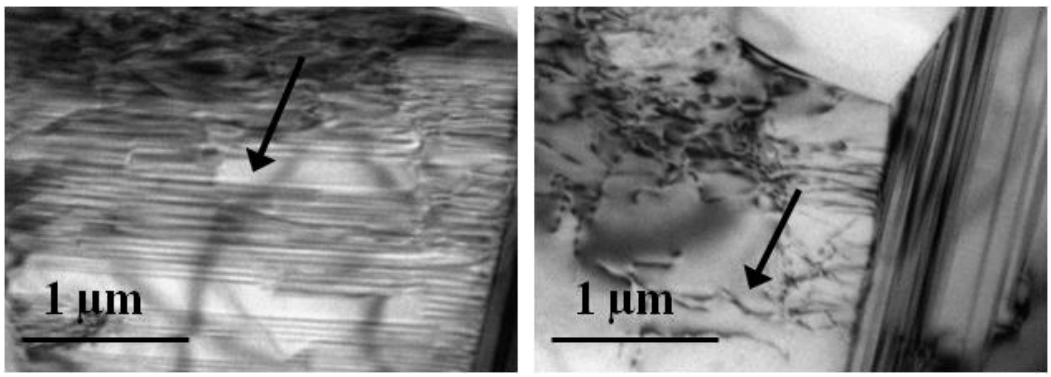

Figure 3. Two TEM images showing stacking faults (a) and dislocations (b), both indicated by arrows.

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