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An Improved TEM Sample Preparation Technique for Heavily Deformed Ceramic Materials 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. Transmission electron microscopy (TEM) is a powerful tool for studying the deformation mechanisms of impacted materials. Indentation allows the study of deformation of microstructural features as a function of distance and depth from the indent. In the original development of the TEM sample preparation technique for observing the structure under indents in SiC used for ceramic armor, Brennan et al. [1] used a multi-step process to preserve the heavily cracked regions under the indent by Tripod polishing, FIB, and vacuum impregnation. Figure 1 outlines the steps of the process developed in that study. In solving the sample preparation problem of heavily cracked ceramic materials, the researchers found several results that are critical for success: 1) vacuum infiltration of a low viscosity epoxy on an exposed cross sectional surface is absolutely required, 2) vacuum infiltration from the top of the surface will not infiltrate the crack structure below the surface but does preserve any spallation from the indentation, 3) mechanical polishing, no matter how fine a grit size is used, closes the paths for the vacuum infiltration and ion milling is required to expose the cracks for infiltration, 4) the FIB lift-out technique cannot be used on the as-indented surface primarily because of re-deposition of amorphous material into exposed cracks, and 5) the epoxy penetration from one surface can be as great as 8 to 10 μm. To minimize ion milling times in the FIB, Brennan et al. aligned the long axis of the Knoop indents along the line of the indents of H-bar samples thinned by Tripod polishing. However, this is not the most favorable orientation to compare the microstructure to fracture mechanics models. Because of the time required to bring the very hard ceramic materials to a thickness of ~50 μm and the two FIB thinning steps, this multi-step technique was a very labor intensive and costly process. The goal of this work was to develop an efficient and less demanding sample preparation technique for heavily cracked ceramics that preserves the true microstructure for TEM imaging and analysis.

Silicon carbide (SiC) and boron carbide (B_4C) were indented using a Knoop indenter with a Tukon 2100B hardness tester. Samples were cut and polished, and then indented with a linear array of constant load indentations. Similar to the previous study, the array of indents were aligned to a surface with care in order to minimize the distance of material to be removed but not affect the indentation fracture mechanics. In the new method, Tripod polishing was eliminated by using a Leica TIC-3X ion milling machine to create the cross sections below the surface by masking approximately half of the indents along the array. After milling, the sample was vacuum infiltrated as in the earlier study. Instead of the final sample having the H-bar configuration, our new procedure uses an in-situ lift-out in the FIB. By removing epoxy on either side of the indent, the surface of the cross section formed by the TIC-3X can be located. This allows the final TEM sample to be made ~2 μm from the TIC-3X prepared surface. Figure 2 shows a low magnification image of a B_4C sample with cross section of four indents visible after milling with the Leica TIC-3X, but prior to infiltration. The insert shows a higher magnification of the indent labeled “2”. Figure 3 shows an SEM image of a vacuum infiltrated sample tilted to an angle of 45° with one FIB lift-out sample with the long axis of the Knoop indents oriented perpendicular to the cross sectional plane. Viewing at 45° allows the indent on the surface to be seen even with the epoxy present.

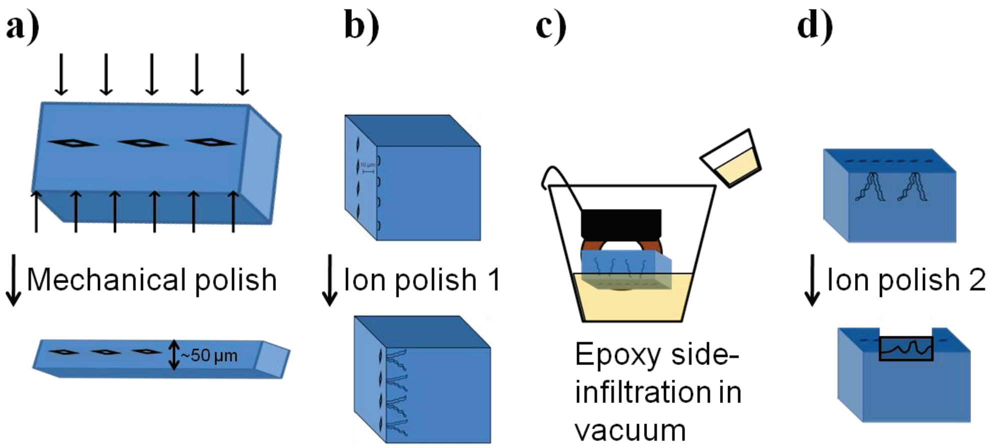

Figure 1. A schematic of the original sample prep process, including mechanical polishing (a), ion milling to open up the cracks (b), epoxy infiltration (c), and final ion polishing (d).

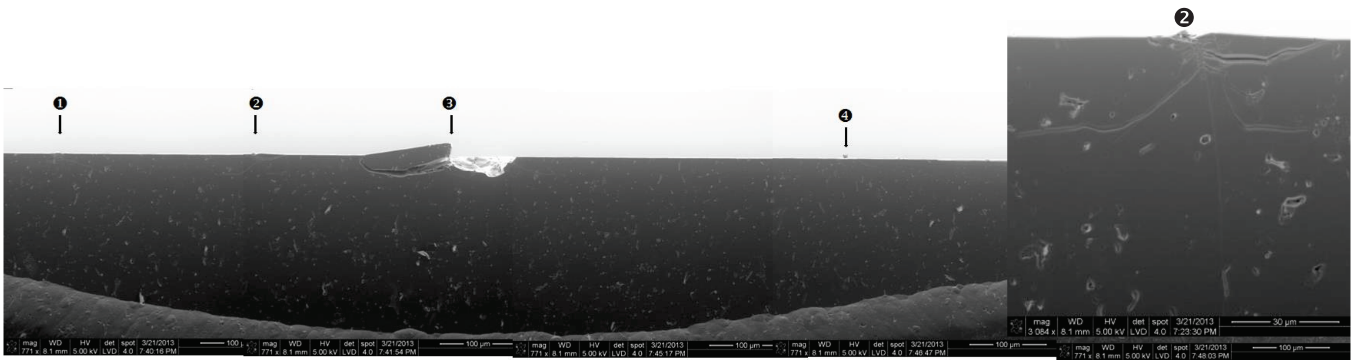

Figure 2. Composite SEM image showing the cross section of indented B4C prepared by Leica TIC-3X prior to vacuum infiltration. Indents are indicated. The inset shows a higher magnification image of “2”.

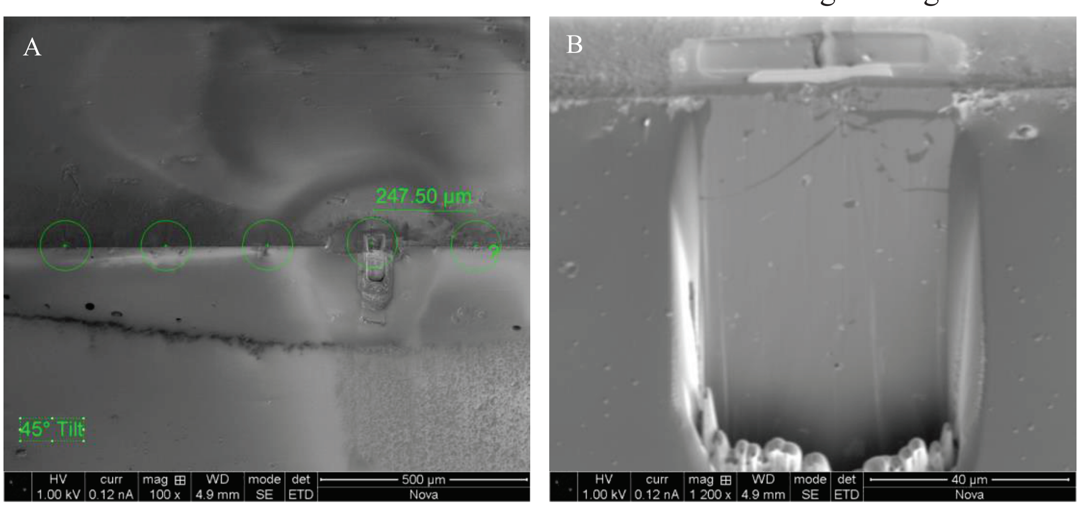

Figure 2. A) 45° tilted SEM image of TIC-3X prepared sample after infiltration and first FIB lift-out area prepared. B) SEM image of indent after epoxy on the cross section surface was removed.

[1] C.V Brennan, S.D Walck, and J. J. Swab, Microscopy and Microanalysis 20(6) (2014), p. 1646-53.