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# TEM Characterization of the Deformed Region Beneath Knoop Indents in Boron Carbide

S. D. Walck[1], J. C. LaSalvia[2], and K. D. Behler[3]

1. TKC Global, RDRL-WMM-C, Aberdeen Proving Ground, MD 21005, USA
2. U.S. Army Research Laboratory, RDRL-WMM-E, Aberdeen Proving Ground, MD 21005, USA
3. TKC Global, RDRL-WMM-E, Aberdeen Proving Ground, MD 21005, USA

Understanding the deformation mechanisms in ceramic materials is crucial for developing and optimizing next-generation ceramic materials in body and vehicle armor systems. In an effort to better understand the mechanistic response of polycrystalline boron carbide to large contact stresses, transmission electron microscopy (TEM) methods were used to examine thin cross-sections of the inelastically deformed regions beneath Knoop indents of various loads and load-dwell times. Indentation allows the study of deformation of microstructural features as a function of distance and depth from the loading and allows for comparison to mechanistic modeling. TEM cross-sections were prepared from 0.3, 1, and 2 kgf Knoop indents in a hot-pressed polycrystalline boron carbide. Due to excessive spallation of material surrounding 2 kgf indents, load-dwell times of 15 and 45 s were only used for the 0.3 and 1 kgf indent loads. Although FIB preparation of nano-indentation methods have been used to examine the areas under indents with great success [1], the higher loads applied in this study allows a more comprehensive examination of the nature of the sub-surface inelastic region and the extended cracking. However, the extensive cracking presents problems for sample preparations that were solved by Brennan et al. using a multi-step sample preparation approach [2]. For the work in this study, an improved technique that was less labor intensive was used [3]. The process involves first preparing the indents in an appropriately spaced array across the surface near the edge, sufficiently close to use a masked-ion milling system (MIMS), but not close enough that the fracture mechanics were compromised. The cross sections of the indents were examined and indexed in an SEM, as shown in Fig 1, and then vacuum infiltrated with a low viscosity epoxy in order to fill the open cracks. The TEM samples were then prepared by in-situ lift-out technique after specific indents were located in the FIB by using the index distances found previously.

TEM characterization of the inelastically deformed regions showed extensive stress-induced amorphization, micro-cracking, and macro-cracking. Observations clearly show that these deformation mechanisms are related. Stress-induced amorphization predominately occurred in discrete slip bands that were nanoscale in width and microscale in length. Micro-cracks were observed at slip band intersections, while macro-cracking appeared to be a consequence of both mechanisms. The trajectory of slip bands appeared similar to slip lines for blunt-wedge indentation, indicating the importance of shear stress on their formation.

References:

[1] K. Madhav Reddy *et al.*, Nature Communications **10**.1038 (2013) p. 3483.
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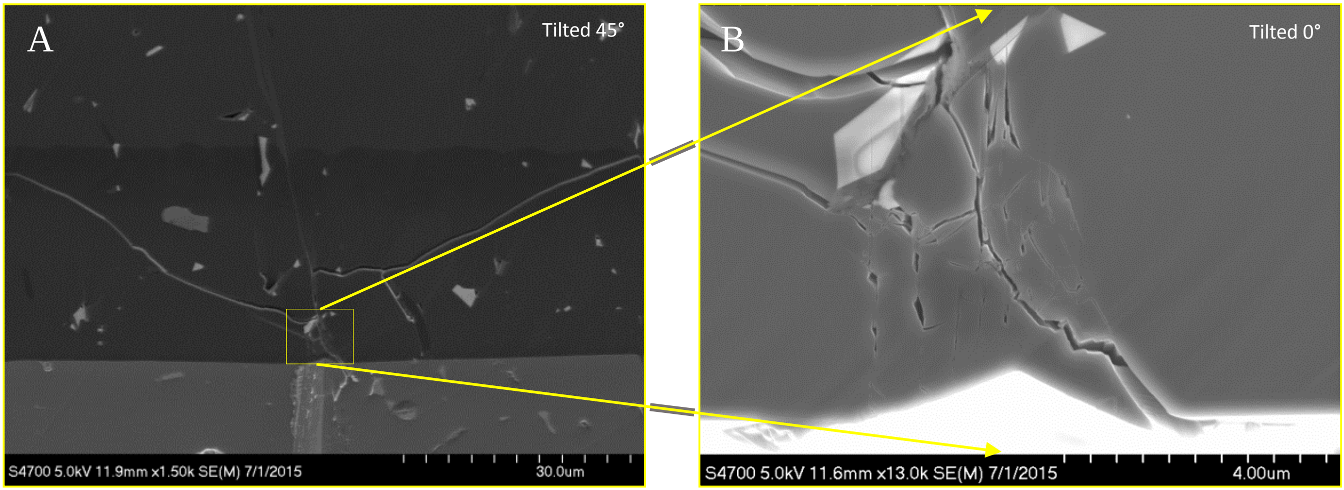


**Figure 1** A)SEM image of MIMS cut sample tilted at 45° showing the cross section and surface of a 1 kgf Knoop indent. B) Enlarged area of (A) showing sub-surface cracking under the indent.

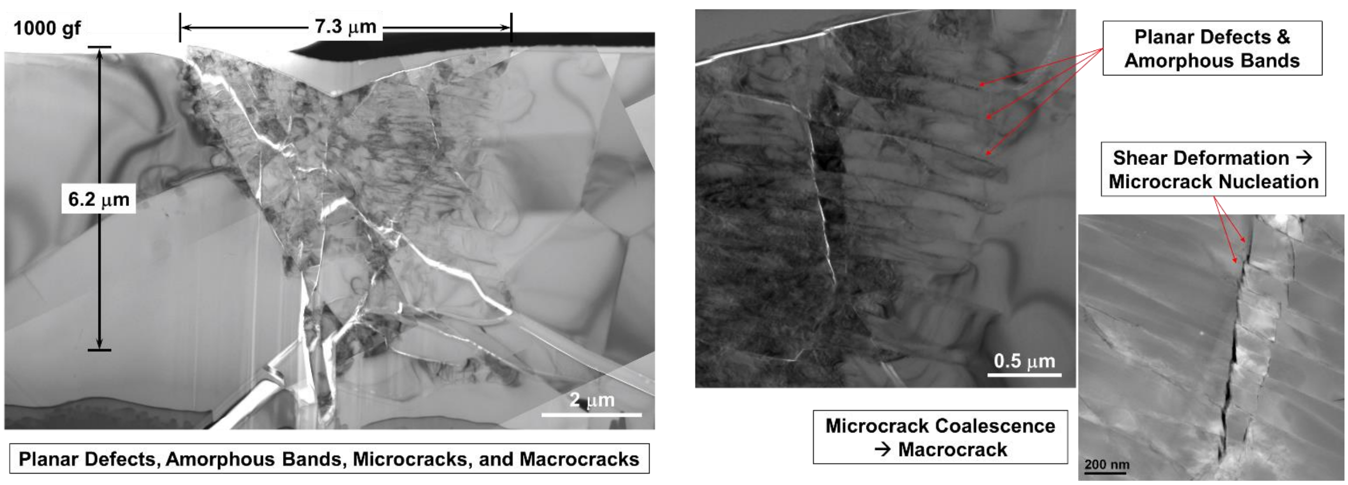


**Figure 2** A) Low Mag XTEM Montage image of 1kgf Knoop indent. B) Enlarged area from (A) showing micro and macro crack formation within the inelastic region under the indent.

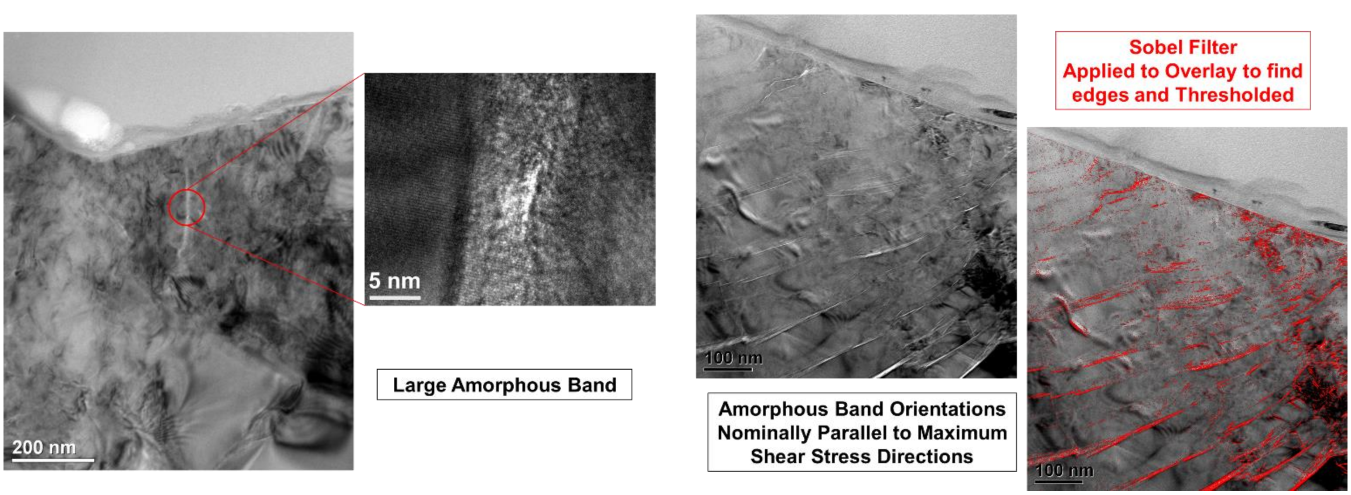


**Figure 3** Inelastic region under a 1kgf Knoop the indent showing the presence and distribution of amorphous shear bands.

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