Floating Zone Crystal Growth and Characterization of Boron Carbide

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Primary PARADIM Tools Used: Laser diode floating zone furnace

Abstract:

Boron carbide is an inexpensive, light weight ceramic with potential for applications in body armor, high temperature thermoelectrical conduction, ionizing radiation shielding, and neutron detection. The rhombohedral crystal structure of the material means that mechanical, electrical, and physical properties may vary along differing crystallographic orientations. Thus, measuring such anisotropic properties requires the use of single crystals. Single crystals of boron carbide are non-trivial to grow; thus, few studies have been conducted measuring the properties of this material across its varying orientations. Single crystals of boron carbide were grown via the floating zone method utilizing PARADIM's laser diode floating zone furnace. From Laue diffraction, we identified several distinct rhombohedral crystal directions, which has allowed and will continue to allow us to measure orientation dependent physical properties.

Summary of Research:

The aim of this project was to determine initial synthetic parameters that may allow for single crystal growth of boron carbide, develop a reliable and reproducible methodology for synthesizing single crystals of boron carbide, identify and isolate multiple distinct crystal planes of the crystal, cut pieces of the crystal along the distinct planes and measure the mechanical, physical, and electrical properties of boron carbide along the different crystal planes.

The anisotropic crystal structure of boron carbide is comprised of icosahedral units that are connected by three atom chains of varying combinations of boron and carbon. There are six distinct crystallographic directions in this rhombohedral structure that have varying mechanical properties (e.g., elastic moduli). These planes are denoted by four coordinate vector directions and can be found using Laue X-ray diffraction.

We utilized a laser diode floating zone furnace to prepare single crystals of boron carbide (m. p. = 2400°C). The laser diodes were used to melt the top portion the seed rod which was then connected to the feed rod in order to establish a molten/floating zone. Once a stable zone was established, both rods were moved downward allowing random boron carbide grains to grow on the seed rod. Over time, several crystal domains begin to dominate the structure of the rod until one orientation is selected. Through this method we were able to produce the longest single crystal of boron carbide reported (about 7.5 cm) as shown in Figure 1. Crystals of larger diameter were recently grown (about 8.3 mm diameter) using a Xenon furnace. Original crystals were 4 mm in diameter. Through Laue diffraction, we were able to identify the [101] and [100] directions of the boron carbide via simulation of their unique symmetry patterns as show in Figure 2.

Once the crystals were grown, they were cut and polished for characterization. Through electron backscatter diffraction (EBSD) we were able to evaluate the orientation of samples cut from the crystal. Transmission electron microscopy (TEM) was used to evaluate to mechanical features of the material. Nanoindentation measurements that use a nano-diamond indenter to apply a stress to a small region of the crystal were conducted. We used powder X-ray diffraction (XRD) to evaluate the materials lattice.

Results and Conclusions:

EBSD results show large crystal domains as shown in Figure 3. TEM results also show the appearance of stacking faults in the crystal as shown in Figure 3. From modeling the XRD data, the a- and c- constants of the crystal lattice were determined to be roughly 5.60Å and 12.1Å respectively. This corresponds to a carbon content of 19.25% to 19.50% based on comparable values found in literature. The results of the nanoindentation tests showed that the hardness of the material across the [101] plane was measured to be 41 GPa as shown in Figure 4. The elastic modulus was measured to be 520 GPa.

We have successfully designed a methodology for the growth of single crystals of boron carbide using the floating zone technique. Indentation and TEM tests have been conducted on samples cut from the single crystal of boron carbide that show results of hardness comparable to that of the known literature [7]. The TEM results also show large domains of single crystals and stacking faults.

Future Work:

Future studies will include the measurements of electrical transport and mechanical properties across different crystallographic directions. These results will be used as a baseline study of the materials behavior in during fracture. Samples will be cut from the large diameter crystals and distributed to our collaborators for use in ballistic performance studies.

Acknowledgements:

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Figure 1: This figure shows the standardly grown single crystal of boron carbide.

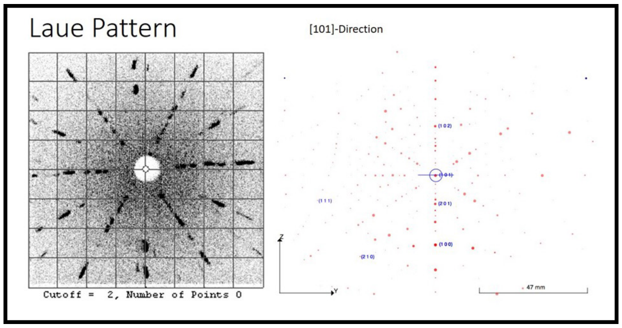

Figure 2: This figure shows the Laue pattern of the [101] direction of the material.

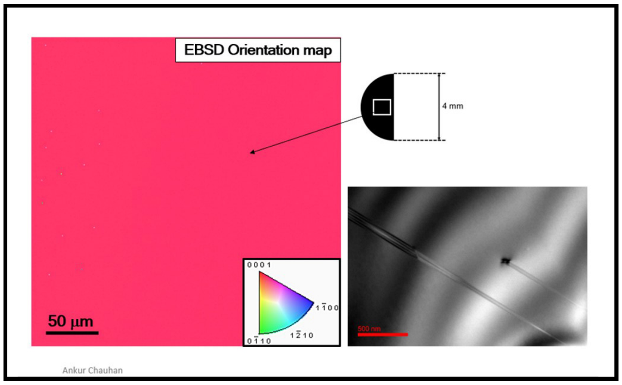

Figure 3: This picture (left) presents the EBSD map of sample showing a single crystal direction. Also (right) it shows the TEM picture of the sample.

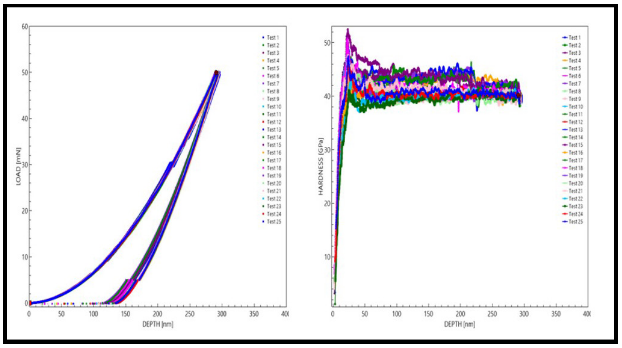

Figure 4: This figure shows the results of the nanoindentation measurements conducted.