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Crystal Growth and Characterization of Boron Carbide

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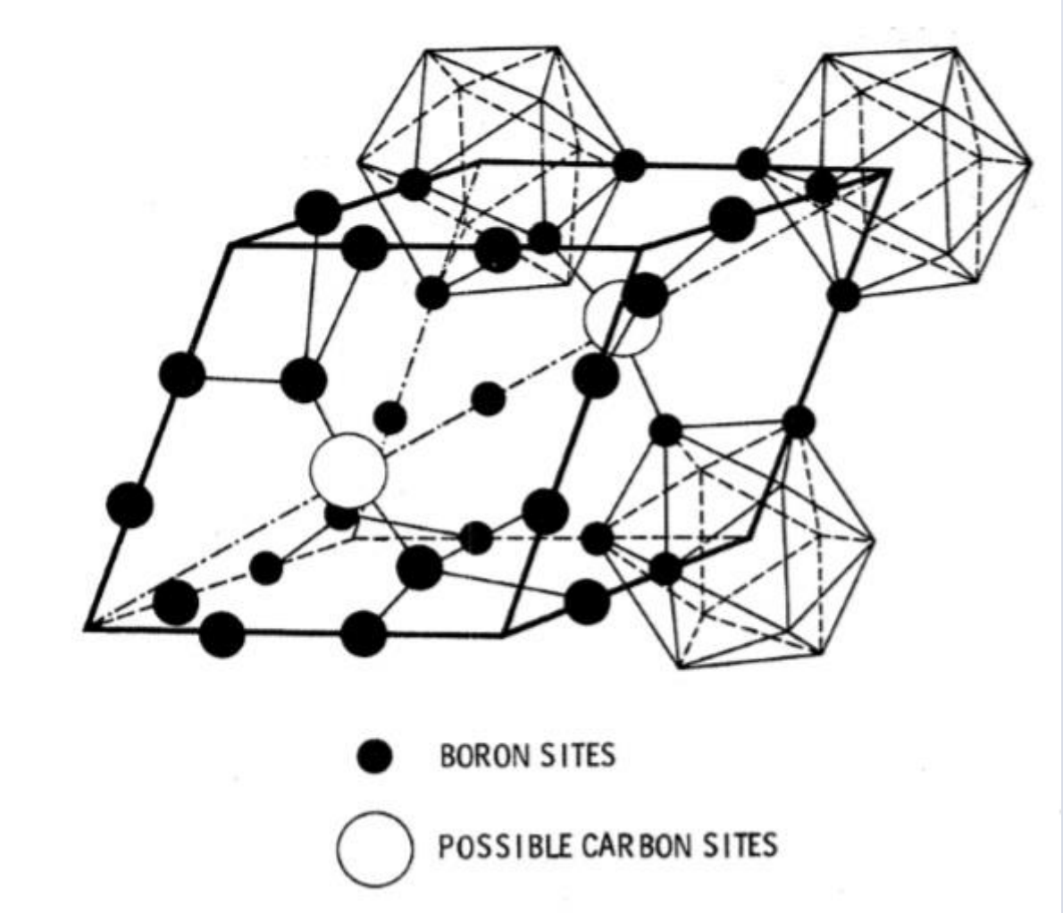
ABSTRACT

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 crystalline structure of the material means that mechanical, electrical, and physical properties may vary along differing orientations. Thus, measuring 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 planes. Single crystals of boron carbide were grown through the floating zone method through utilizing PARADIM"s laser diode floating zone furnace. Using laue diffraction, we identified several distinct rhombohedral crystal directions. The material was then cut along these planes using multiple methods which include a diamond crystal cutter saw and a diamond wire saw. In future work these slices of the crystal will be used for impact testing, transport properties measurements, etc.

OBJECTIVE

- Grow single crystal of boron carbide.
- Develop a reliable and reproducible methodology for creating single crystals of boron carbide.
- Identify and isolate multiple distinct crystal planes of the crystal.
- Cut pieces of the crystal along the distinct planes.
- Measure the mechanical, physical, and electrical properties of boron carbide along the different crystal planes.

CRYSTAL STRUCTURE & CHARACTERISTICS



(Wood & Emin, 1984)

- The crystal structure of boron carbide is comprised of icosahedral units that are connected by three or two atom chains that are comprised of varying combinations of boron and carbon.
- In crystallography there are six distinct crystal planes that are intrinsic of crystals that are structured in this rhombohedral shape. These planes are denoted by four coordinate vector directions and can be found using Laue x-ray diffraction.

FLOATING ZONE TECHNIQUE

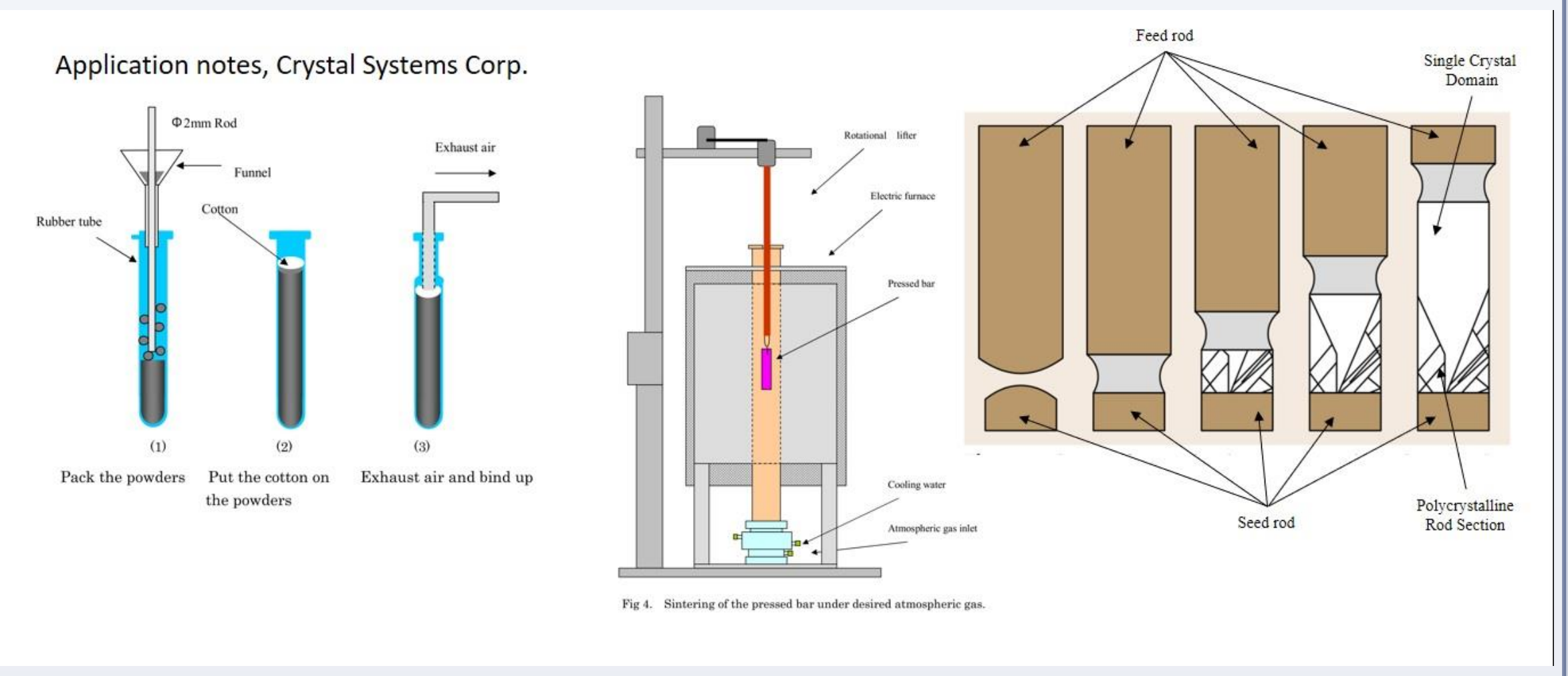

Application notes, Crystal Systems Corp.

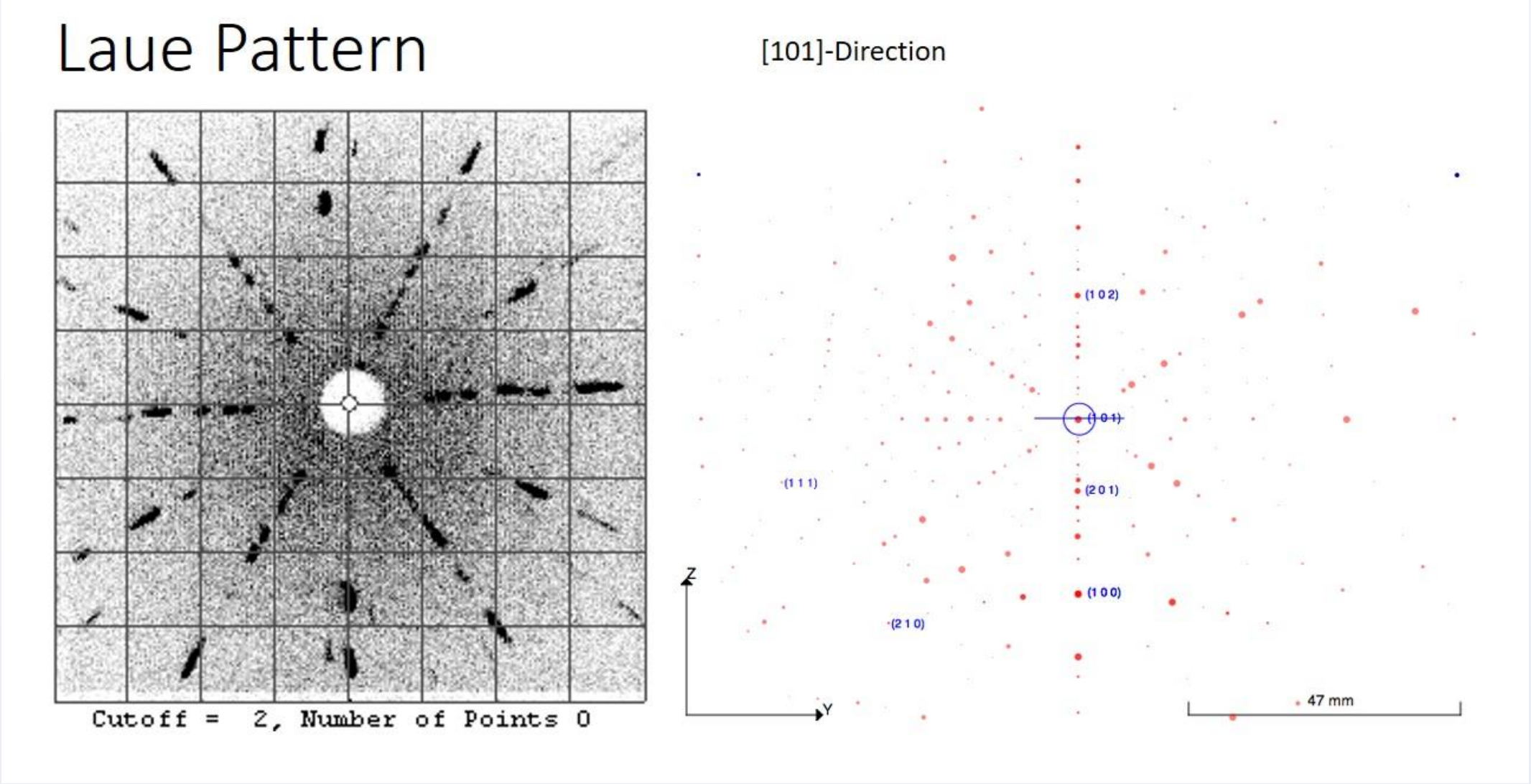
Fig 4. Sintering of the pressed bar under desired atmospheric gas.

- We utilized a laser diode floating zone furnace to create single crystals of boron carbide (m. p. = 2400ºC).
- In the floating zone crystal growth method, powder of the material is packed into balloons that are then pressed into single consolidated rods.
- 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 zone. Once a stable molten zone is established, both rods are moved downward allowing the molten zone to travel up the seed rod. As the material solidifies it becomes part of a single rod of boron carbide. Over time, several crystal domains begin to dominate the structure of the rod until one orientation is selected. This becomes the single crystal portion of the rod.

- Through the use of this method we were able to produce the longest single crystal of boron carbide reported (about 7.5cm).

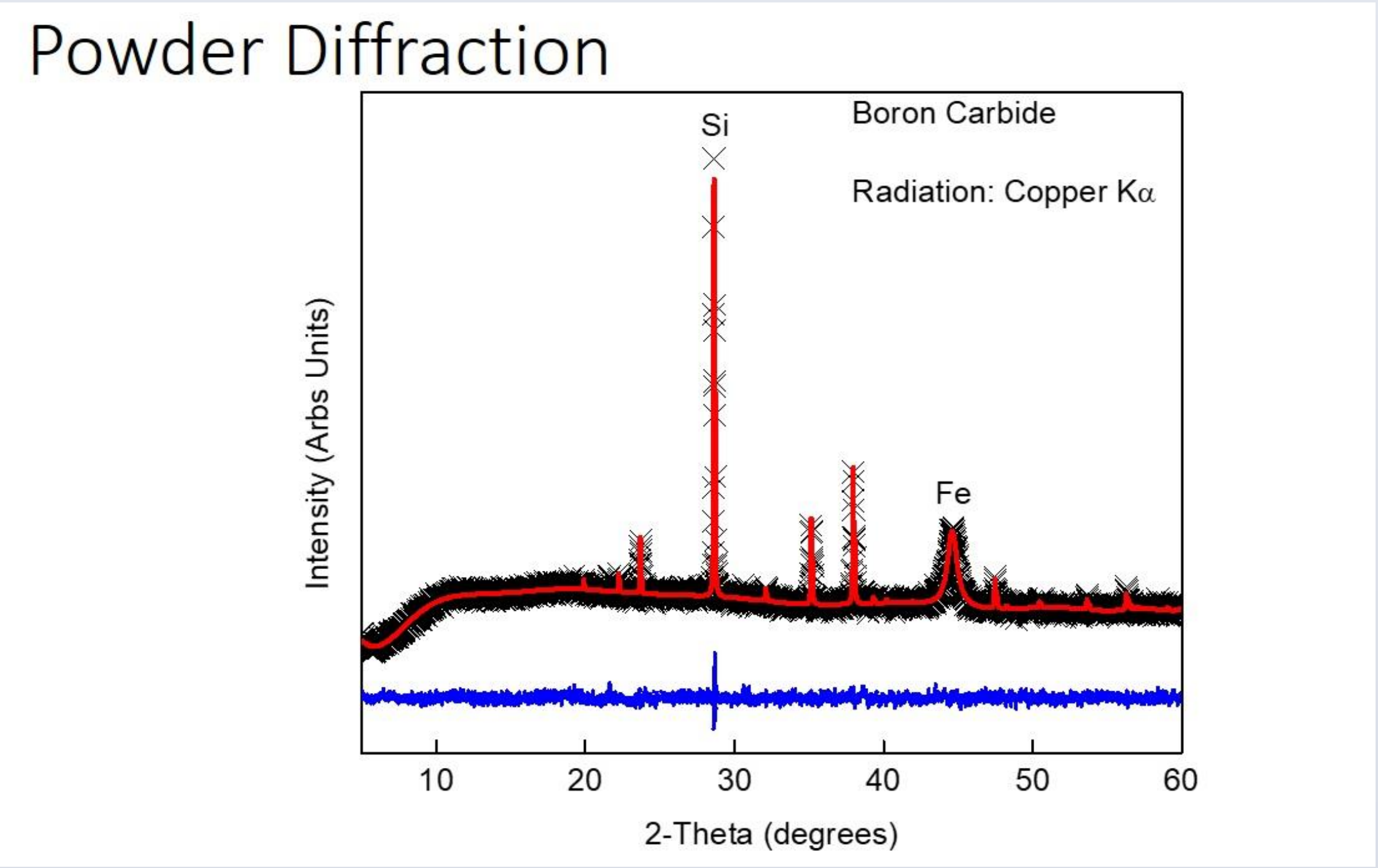
- Graphitic skin was observed pealing away from the molten zone during the growth process.
- Using this technique multiple times on a single sample purifies the crystal, as the impurities are pulled toward the solid-liquid barrier while molten.
- To verify that product of this growth technique was a single crystal, the crystal was placed on a goniometer and evaluated using Laue Diffraction.

LAUE DIFFRACTION



- In Laue diffraction, collimated multi-wavelength x-rays are fired at a material and diffracted back by the atoms. The diffraction pattern is measured by the detector in the device.
- Through the use of Laue Diffraction, we were able to identify the 101 direction of the boron carbide. The 101 crystal direction is marked by its three bisecting leaflets and symmetry across each bisecting line.
- We were also able to locate and image the 001 crystal direction of the sample.

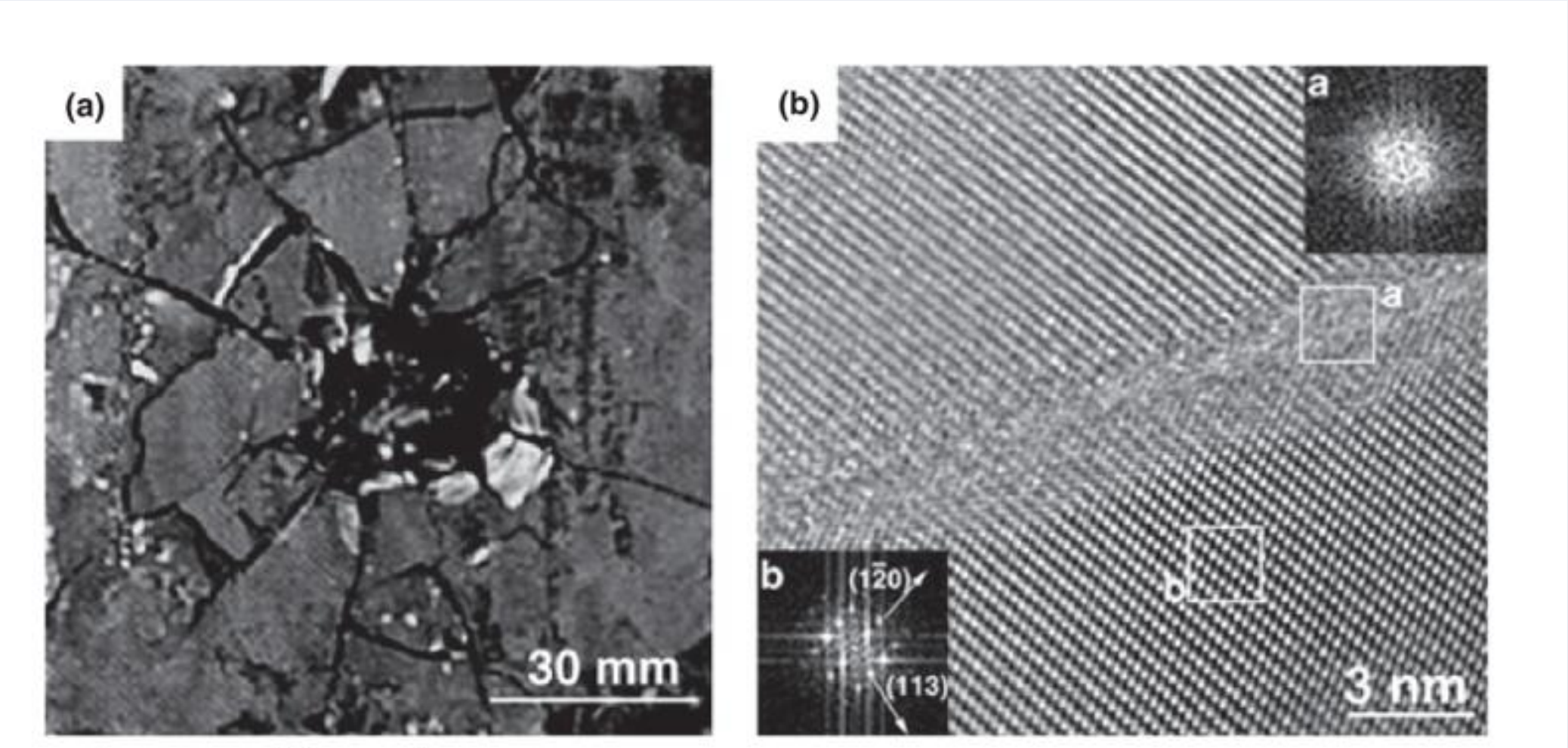
POWDER X-RAY DIFFRACTION



- In powder x-ray diffraction, x-rays of a single wavelength are fired at a sample that is crushed and plated. The x-ray scatters back at a detector but only at specific angles. The intensity and angles at which the x-rays diffract back can be used to interpret the lattice positions/atom types and unit cell type of the crystal, respectively.
- A silicon standard was added to the boron carbide powder sample as a standard to adjust for shifts in the data.
- In the results shown in the figure above, there is a large peak that represents the silicon but also a large peak in the right half of the figure that is representative of iron in the sample. This iron comes from the steel mortar and pestle used to ground the extremely hard boron carbide into powder. Using Le Bail fit, we have determined that the crystals grown are pure boron carbide.

DISCUSSION AND FUTURE DIRECTIONS

- We have successfully grown several single crystals of boron carbide using the floating zone technique which establishes it as a reproducible method.
- Indentation tests will be conducted on samples cut from the single crystal of boron carbide to use as a baseline study of the materials behavior in during fracture.
- Other future studies will include the measuring the transport of the material. This will be done by cutting a piece of the sample into a bar and painting leads on it. This will be performed using a physical properties measuring system.



Chen et al (2003)

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REFERENCES

1) Balakrishnarajan, M., Pancharatna, P., & Hoffmann, R. (2007). Structure and bonding in boron carbide: The invincibility of imperfections. *New Journal Of Chemistry*, *31*(4), 473. doi: 10.1039/b618493f
2) Hushur, A., Manghnani, M., Werheit, H., Dera, P., & Williams, Q. (2016). High-pressure phase transition makes B4.3C boron carbide a wide-gap semiconductor. *Journal Of Physics: Condensed Matter*, *28*(4), 045403. doi: 10.1088/0953-8984/28/4/045403
3) Wood, C., & Emin, D. (1984). Conduction mechanism in boron carbide. *Physical Review B*, *29*(8), 4582-4587. doi: 10.1103/physrevb.29.4582
4) X RAY DIFFRACTION. (2018). Retrieved from https://sseaimes.wordpress.com/2011/08/23/x-ray-diffraction-2/
5) Domnich, V., Reynaud, S., Haber, R., & Chhowalla, M. (2011). Boron Carbide: Structure, Properties, and Stability under Stress. *Journal Of The American Ceramic Society*, *94*(11), 3605-3628. doi: 10.1111/j.1551-2916.2011.04865.x
6) Chen, M. (2003). Shock-Induced Localized Amorphization in Boron Carbide. *Science*, *299*(5612), 1563-1566. doi: 10.1126/science.1080819