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X-ray diffraction study of superhard BN phases synthesized in a rotational diamond anvil cell

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Beamline(s): X17C, X17B

Introduction: The high-pressure behavior of boron nitride (BN) has been an interesting topic because of the quenchable superhard phases, i.e., cubic BN (cBN) and wurtzite type BN (wBN). Transformation properties have been extensively studied using multi anvil and shock wave techniques. However, the studies by a diamond anvil cell, particularly x-ray diffraction studies have been very difficult because both boron and nitrogen are low Z element, and have low scattering coefficients. Moreover, the conventional diamond anvil cell mostly concentrates on the hydrostatic compression. Behavior of BN under non-hydrostatic conditions has not been well studied. The Rotational diamond anvil cell (RDAC) has the capability to superimpose rotation during compression, which can therefore generate shear strain under high pressures. In this study we report our synchrotron x-ray diffraction study of RDAC processed BN.

Methods and Materials: We used a RDAC in our sample processing. A stainless steel gasket with a 250 μm diameter hole, and initial thickness of 212 μm was adopted. Pressure was increased to 10 GPa, at which point the thickness of the sample was measured to be 58 μm. Plastic sheer was induced at this point, into the sample by rotating the diamond anvil by approximately 120^{o} (60^{o} CCW followed by 60^{o} CW). The thickness was dropped to approximately 24 μm. No discernable additional enlargement of the hole was observed at this point. Pressure was then increased to 25 GPa with a measured sample thickness of 11 μm. The sample was subjected to plastic shear by rotating the anvil in both directions for a total amount of 240^{o}. The thickness of the sample reduced to 5 μm after this rotation stage. Fig.1 shows the sample before and after the process.

Synchrotron x-ray diffraction was carried out at beam lines X17C and X17B by using the energy dispersive method. The beam size is confined to 25X30 micron. Different 2-theta was chosen to eliminate to fluorescence and escape lines. Sample was measured in different spots along the radial direction with a 30-micron step. A 360 degree chi-rotation was performed during the measurements at the center position of the sample.

Results: Fig. 2 shows the typical x-ray diffraction pattern obtained from the center position. This pattern can be well indexed into a wBN, in accordance to reference [1]. The main exception is that broad band at about 2.1 angstrom, which can be fitted into two peaks, one at indexed to (002) of wBN, and the other matched that of (111) of cBN. We found strong diffraction peaks at the (220) and (311) position. These peaks, however, cannot be distinguished from the (110) and (112) of wBN, respectively. In the figure, we also marked the hBN position. Although we cannot clearly distinguish any of the hBN lines from the pattern, we still cannot exclude the existence of this phase because of its possible weak diffraction and overlapping with other two phases.

The patterns obtained along the radio direction are consistent with each other. Therefore the sample as a whole has undergone an identical transformation.

Conclusions: We conclude that the hBN has transformed to a mixture of wBN and cBN under the combined compression and shearing process. Since wBN has not been observed under hydrostatic compression at such a pressure, this process provides us with a unique technique for high pressure synthesis.

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References: 1. Soma. T et al, Mater. Res. Bull., 9, 755(1974).
2. Cannon, J. Humphreys, Brigham Young University, Provo. Utah, USA, ICDD Grant-in-Aid, (1983).

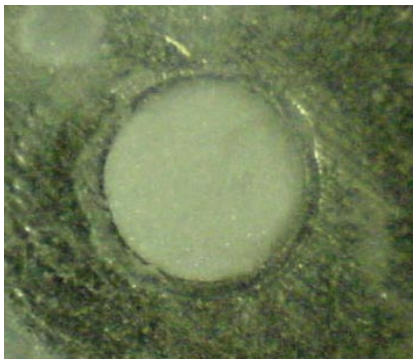

Fig. 1. Sample in a gasket before and after the process
Left: Hexagonal BN (white powder) in a stainless steel gasket prior to loading, Right: The resulting mixture of cBN and wBN (dark brown color) after pressurization and shear in the RDAC.

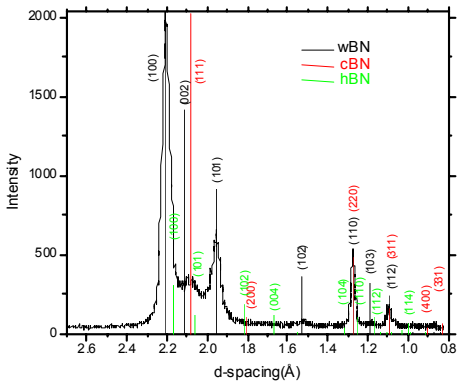

Fig. 2 X-ray diffraction pattern of the high-pressure and shear processed BN
(cBN pattern from Ref. 2, with sample from GE.)