SUPPLEMENTARY INFORMATION: DETERMINING CRYSTAL PHASE PURITY IN *c*-BP THROUGH X-RAY ABSORPTION SPECTROSCOPY

This document contains supporting informating for the publication entitled “Determining crystal phase purity in *c*-BP through x-ray absorption spectroscopy” by S.P.Huber *et al.* with DOI: 10.1039/C6CPxxx

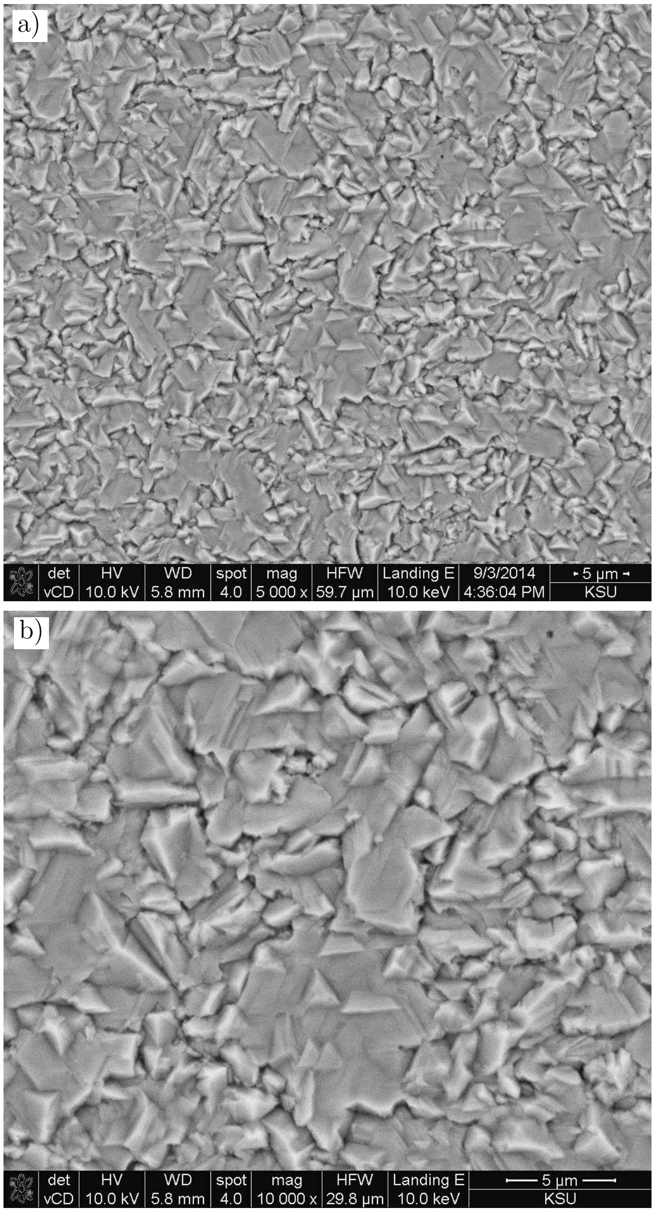

FIG. S1. SEM micrograph of sample S1 at (a) 5000 × and (b) 10 000 × magnification. The micrographs reveal a polycrystalline structure with a strong texturing.

A. I. Scanning Electron Microscopy

The surface morphology of sample S1 was characterized by scanning electron microscopy (SEM) using an FEI Versa 3D DualBeam microscope. Micrographs taken at two different zoom levels for sample S1 are shown in Fig. S1. The micrographs reveal that the sample surface has a strongly textured morphology and a polycrystalline structure. The grains vary in size and the crystallites tend to have triangular features, but do not necessarily all seem to be oriented in a specific direction. Micrographs of sample S2 are not available unfortuntaly, but samples grown under comparable conditions, show that at reduced deposition temperatures, the grains are smaller in size and tend to have lesser defined geometrical features and crystalline orientations[S1].

B. II. X-ray Diffraction

The crystalline nature of samples S1 and S2 has been assessed by x-ray diffraction, using a Panalytical Empyrean diffractometer with Cu-Kα_1 radiation ($\lambda = 1.5406$ Å). Spectra were recorded in thin film geometry, i.e. 2θ scans at various fixed incident angles, as well as Bragg Brentano geometry (coupled $\omega - 2\theta$). For the latter, the diffractometer was aligned such that the momentum transfer axis was aligned perpendicular to the SiC (004) planes, introducing an offset to the $\omega - 2\theta$ scans with respect to the sample surface. This enables one to assess whether during BP growth, any BP crystallite planes have a preferred orientation with respect to the crystal orientation of the SiC substrate.

From the thin film XRD scans of samples S1 and S2, shown in Fig. S2(a) and S2(c) respectively, it is immediately clear that the samples have a strong texture. For both samples, the BP diffraction peaks do not show a typical spectrum consistent with powder reference spectra as only a few of the expected preferential diffraction angles are visible. The textured geometry was confirmed by subsequent rocking scans (fixed 2θ, with ω as scan axis) which show diffracted intensity only at the incident angle that matches the offset of the SiC substrate. The measurement taken in Bragg Brentano geometry for samples S1 and S2 is shown in Fig. S2(b) and S2(d) respectively. The single weak peak at 24.8° in Fig. S2(d), which corresponds to a 2θ of 57.5°, given the 4° miscut of the substrate, confirms that sample S2 has strong texturing with the (220) faces of the *c*-BP crystal having a preferred orientation parallel to the (400) SiC planes. A similar conlusion can be drawn for S1, which has a preferred orientation of the (111) faces with respect to the substrate, as made evident by the strong peak at ∼21° in the rocking curve shown in Fig. S2(b). The main difference between the two samples appears to be that the textured nature is much stronger for samples S1, which could be due to a more developed crystal structure for this, much thicker, sample.

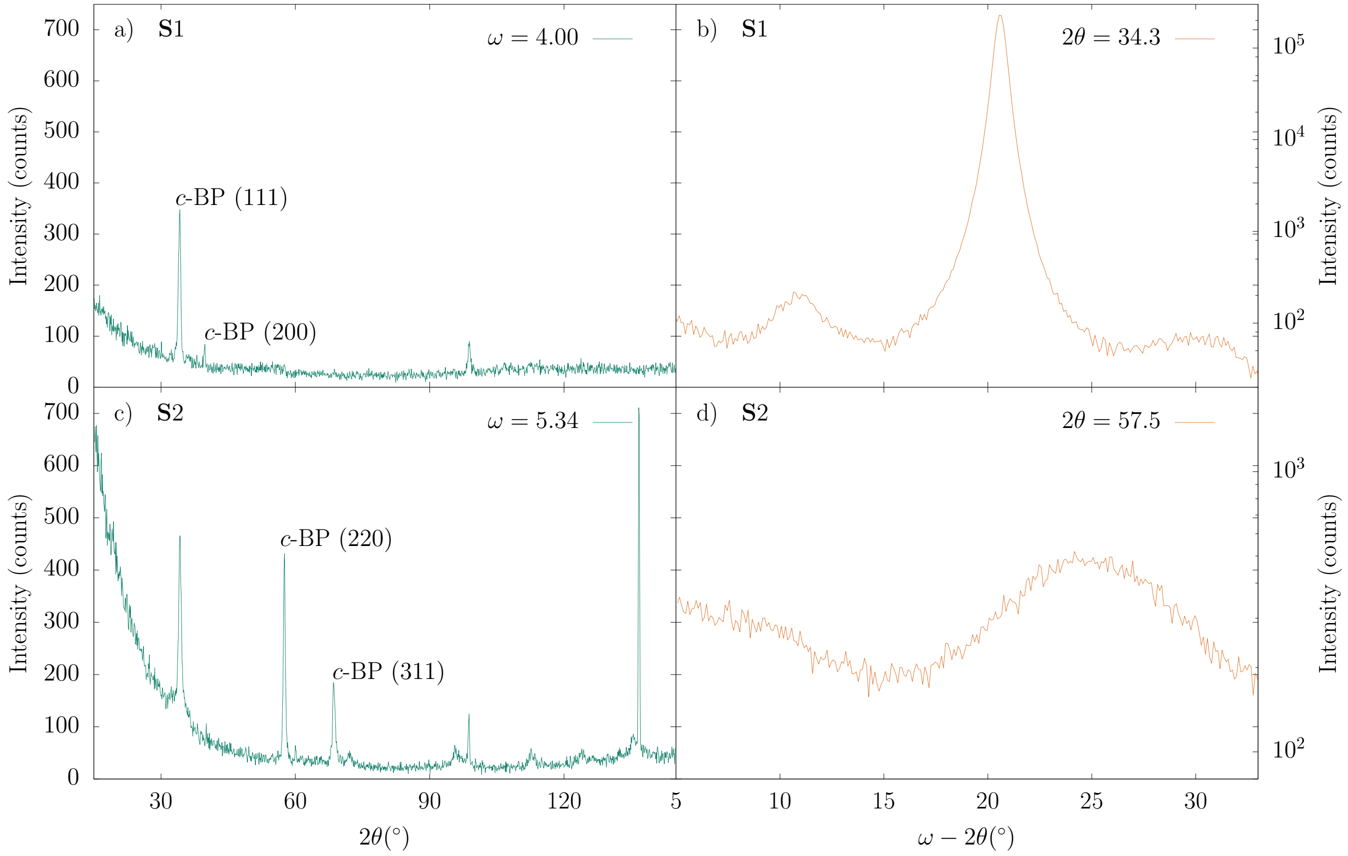

FIG. S2. (a,c) X-ray diffraction spectra in thin film geometry for samples S1 and S2, respectively, at different values of ω. The labels at the peak denote the corresponding *c*-BP planes taken from powder reference spectra. (b,d) Rocking scans in Bragg Brentano geometry for samples S1 and S2, respectively, with different fixed values of 2θ.

Due to the strong texturing in both samples and their thin film nature, it is impossible to quantitatively assess their absolute or even relative crystallinity from this data. It is therefore also impossible to definitively prove the presence of any amorphous phases in the samples based on the XRD analysis. The combined SEM and XRD data shows that both samples exhibit a polycrystalline morphology, however, the hypothesis of the presence of amorphous phases, while certainly compatible and likely, cannot be decisively proven nor debunked.

[S1] B. Padavala, C. Frye, Z. Ding, R. Chen, M. Dudley, B. Raghothamachar, N. Khan, and J. Edgar, Solid State Sciences **47**, 55 (2015).