Quantification of Hexagonal Boron Nitride impurities in Boron Nitride Nanotubes via FT-IR Spectroscopy

Supplemental Information

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Figure SI 1. FT-IR transmission spectra of boron oxide overlaid on as received BNNTs and h-BN.
Figure SI 2. Schematic diagram of ultrasonically assisted diffusion separation of BNNTs and h-BN.

Figure SI 3. TEM micrographs of BNNT samples. From Left to Right, as-received BNNTs (AR-BNNTs), oxidized and washed BNNTs (OW-BNNTs), and Triton X-100 separated BNNTs (TX-BNNTs). The red scale bar for each image is 200 nm.
Useful Literature Images Provided for Analysis Perspective

Figure SI 4. FT-IR transmission spectrum from highly pure boron nitride nanotubes (BNNTs). Reproduced from Chee Huei et al. Vibrational modes along the tube axis (LO) of a BNNT correspond to $\sim 1369 \text{ cm}^{-1}$, while circumferential or tangential vibration modes (T) tangent to the axis are at $\sim 1545 \text{ cm}^{-1}$. Out-of-plane radial buckling modes are at $\sim 809 \text{ cm}^{-1}$.

Figure SI 5. HRSEM image from Okan et al. Figure 9b sample BNNT-3 & Figure 9d sample BNNT-5. Representative SEM images of the BN structures formed during the synthesis described in the study.
Boron Oxide and Boric Acid

Figure SI 6. XRD patterns of a) boric acid and b) boron oxide powder after calcination of boric acid in Ar\textsubscript{(g)} at 400 °C.\textsuperscript{2} A notable observation is the presence of a 2θ diffraction near 27°, which has been assigned to boron oxide (b) but overlaps similar peaks for boric acid (a), and peaks commonly assigned to (002) h-BN in many studies (Figures 6 and 7 of the main text).

Hexagonal Boron Nitride (h-BN)

Figure SI 7. XRD Spectra of pure hexagonal boron nitride (h-BN), reproduced from Figure 3a and 3b in Kumari et al.\textsuperscript{5} a) The diffraction peaks corresponding to the (002), (100), (101), (102), (004), and (110) planes are all clearly shown and is characteristic of highly ordered h-BN structure. b) The (002) peak is symmetric and narrow at a 2θ = 26.75° with a FWHM of 0.36°.
Figure SI 8. XRD spectra reproduced from Thomas et al.\textsuperscript{6} and Figure 1 in Balint et al.\textsuperscript{3}
Resolution of the characteristic peaks of h-BN increase in order from A – D; showing the effects of three-dimensional ordering, from turbostratic boron nitride to complete three-dimensional order, respectively.
Figure SI 9. a) XRD patterns of BN nanostructures produced using pure boron powder. Diffraction peaks of (002) h-BN are observed at $2\theta = 27.7^\circ$, 28°, and 28.4° for BNNT-1, BNNT-2, and BNNT-3, respectively. A diffraction peak of (100) h-BN is observed at $2\theta \approx 41^\circ$ in BNNT-1. $\text{B}_2\text{O}_3$ is observed in BNNT-1 at $2\theta \approx 14^\circ$, while unreacted Boron in BNNT-1 and BNNT-2 is observed between $2\theta = 15^\circ - 25^\circ$. b) A diffraction peak of Fe-B, a high temperature (750 °C) side product appears at $2\theta \approx 27^\circ$ for BNNT-5, compared to BNNT-1 produced at a lower temperature (600 °C).
Figure SI 10. XRD patterns of BNNTs i-iii) as-synthesized, dispersed, and acid washed, respectively; iv-vi) oxidized at 750 °C, 800 °C, and 850 °C, respectively; vii) sample (v) washed with hot water. Typical diffraction peak of (002) h-BN appears around 2θ ≈ 31° in all samples, and B₂O₃ is observed at 2θ ≈ 16° for oxidized samples.

Figure SI 11. XRD spectra of BNNTs a) before oxidation, b) after oxidation at 700 °C (air), and c) further oxidized in air up to 900 °C. Peak designations are X: Fe; ○:Fe₂O₃ and *: B₂O₃, although not including 2θ angles below 20° in the spectra, to show the 2θ ≈ 15° formation of boron oxide is a crucial omission from this data.


