SUPPORTING INFORMATION

Synthesis of Highly Crystalline sp²-Bonded Boron Nitride Aerogels

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This document contains additional microscopy images, graphics and discussion of additional characterization (XRD and nitrogen adsorption isotherms) of the BN aerogels discussed in the text, as well as a schematic detailing the crucible in which the synthesis of the aerogels is performed.



Figure S1: Additional TEM images of precursor graphene aerogels. Scale bars are 20 nm (upper left) and 10 nm (all others)



Figure S2: Additional TEM images of converted sp²-bonded BN aerogels. Scale bars are 50 nm (upper left), 500 nm (upper right), 20 nm (lower left), and 10 nm (lower right)



Figure S3: XRD spectrum of a BN aerogel. Expected peak positions for hexagonal (blue) and rhombohedral (green) BN. Substrate peaks are marked with an asterisk. Owing to the low density of our material, few atoms are available for scattering in the target region of the instrument, leading to weak diffraction peaks. Nonetheless, the peak at 26.5° corresponds to an interplanar spacing of $0.337 \pm .009$ nm, consistent both with accepted values of sp²-bonded BN and those measured in our TEM images. The sample contains higher angle peaks from patterns of both hexagonal and rhombohedral BN. The less common rhombohedral BN has also been found in previous studies of BN synthesized *via* carbothermal reduction of boron oxide.¹



Figure S4: Additional examples of junctions and cross-links between sheets in BN aerogels. Scale bars are 10 nm.



Figure S5: Nitrogen adsorption isotherms for graphene and BN aerogels. Both adsorption curves exhibit hysteresis Type H3 under the IUPAC classification,² but the size of the hysteresis loop for the BN aerogel is noticeably smaller. This could indicate the disappearance of mesopores by the straightening of the aerogel sheets upon conversion to BN. It should be noted that small sample sizes limit somewhat our porosimetry results.



Figure S6: Schematic of conversion crucible for carbothermal reduction of graphene aerogels in nitrogen. Red arrows indicate nitrogen flow, blue arrows indicate rising boron oxide vapor, and purple arrows indicate a mixture of these two gases. The sample is placed in the perforated cup in the center of the crucible and heated in an induction furnace. All parts are machined from high density molded graphite. After a few conversions, all interior surfaces of the crucible are converted to boron nitride. The outer diameter of the crucible is 5 cm.

REFERENCES for SUPPORTING MATERIAL

- 1. Bartnitskaya, T. S.; Kosolapova, T. Ya.; Kurdyumov, A. V; Oleinik, G. S.; Pilyankevich, A. N. Structure and Some Properties of Fine-Grained Graphite-Like Boron Nitride. *J. Less-Common Met.* **1986**, *117*, 253–258.
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