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Supporting Information

Nonconductive Layered Hexagonal Boron Nitride Exfoliation by Bipolar Electrochemistry

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Experimental section

Materials.

Boron nitride was purchased from Merck (USA). Sodium sulfate, sodium phosphate monobasic, potassium phosphate dibasic, potassium chloride and sodium chloride were purchased from Sigma-Aldrich. Pt, Ag/AgCl reference, and glassy carbon electrodes were purchased from CH instruments, Texas, USA. Deionized water was used in the preparation of solutions.

Apparatus.

Transmission electron microscopy (TEM), selected area electron diffraction (SAED) and high resolution transmission electron microscopy (HR-TEM) were performed using EFTEM Jeol 2200 FS microscope (Jeol, Japan), a 200 keV acceleration voltage was used for measurement. Sample preparation was attained by drop casting the suspension (1 mg mL^{-1} in water) on a TEM grid (Cu; 200 mesh; Formvar/carbon) and dried at 60 °C for 12 h. Elemental maps and energy dispersive spectrometer (EDS) spectra were acquired with SDD detector X-MaxN 80 T S from Oxford Instruments (England). Scanning electron microscopy and scanning transmission electron microscopy (STEM) were performed with a JEOL 7600F field emission SEM (JEOL, Japan). SEM and STEM images were obtained at an acceleration voltage of 2 KV and 30 kV, respectively. X-ray photoelectron spectroscopy (XPS) was conducted using an X-ray photoelectron Phoibos 100 spectrometer (SPECS, Germany), equipped with a monochromatic Mg Kα X-ray radiation source at 1253 eV. Graphite was added to XPS measurement in order to obtain C 1s peak for calibration. Raman spectra were obtained by using a confocal micro-Raman LabRam HR instrument (HORIBA Scientific) in backscattering geometry with a CCD detector, a 514.5 nm Ar laser and a 100X objective attached to an Olympus optical microscope. Atomic force microscopy (AFM) conducted on NaioAFM microscope (Liestal, Switzerland) fitted with an ACL-A cantilever tip (AppNano, CSInstruments, France) with the following dimensions: length, 225 µm; width, 43 µm; tip radius, 6 nm. The NaioAFM microscope was connected to a computer with Nanosurf, Naio control software (version 3.8.0.8) and Nanosurf, easyScan 2 control software. The AFM images were obtained when the microscope was operated under dynamic force at vibration amplitude of 200 mV. The AFM image size of 1.0 µm by 1.0 µm was acquired. The AFM is mounted on an airtable TS-150 (Herzan, California, USA) for all measurements. Electrochemical experiments were carried out at room temperature by using an AutoLab PGSTAT 101 electrochemical analyzer (Eco Chemie, Utrecht, The Netherlands), controlled with a NOVA version 1.10 software (Eco Chemie).

Electrochemical exfoliation of *h*-BN.

Exfoliation of *h*-BN was conducted following the same protocol established by our group.[27] 5.0 mg ml^{-1} of aqueous suspension of *h*-BN was prepared in deionized water by sonication for 3 hours. This homogeneous *h*-BN suspension was diluted to 0.5 mg ml^{-1} by adding deionized water and 0.5 M Na_2SO_4 with the total volume of 4 ml and placed into an electrochemical cell. Two platinum driver electrodes were immersed into the electrochemical cell and 10 V potential was supplied for 30 min. After that, the suspensions were left to settle for a minimum of 48 h, and the solution was then centrifuged with the speed of 14000 rpm for 15 min and washed with deionized water for several times in order to remove Na_2SO_4 for characterization.

Electrochemical measurement of inherent electrochemistry.

0.5 mg mL^{-1} concentration of bulk *h*-BN and *h*-BN nanosheet was prepared in deionized water. Glassy carbon electrode was renewed by polishing with a 0.05 µm alumina particle slurry on a polishing pad for 1min and then rinsed with deionized water and dried. Next, 4 µL aliquot of material suspension was drop casted on the electrode surface, which are dried at room temperature in order to get homogeneously distributed materials on the electrode surface. Cyclic voltammetry measurements was performed in a three electrode system where Pt, Ag/AgCl and glassy carbon as counter, reference and working electrode. Phosphate buffer solution (PBS, 0.5X10^{-3} M, pH 7.2) was utilized as background electrolyte which was purged with N_2 gas for 15 minutes and all cyclic voltammetry was performed at 100 mV s^{-1}.

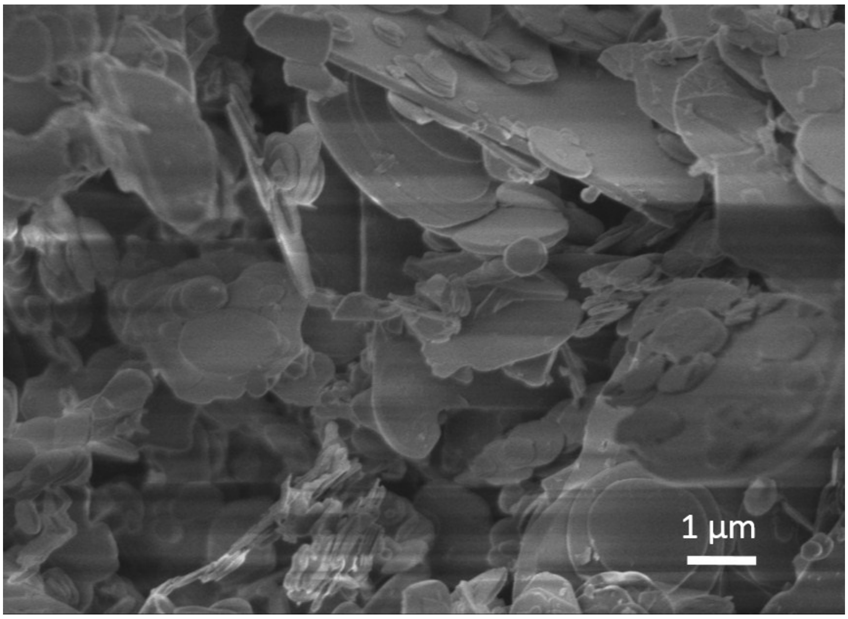

Figure S1. Morphological characterization: SEM image of bulk *h*-BN.