

Supporting information for

**A Solvent Decomposition and Explosion Approach for Boron Nanoplates Synthesis**

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**Experimental Section**

*Synthesis of surface functionalized boron nanoplates:*

1 g NH<sub>4</sub>Cl (99.5%, Aladdin) and 50 mg MgB<sub>2</sub> (99%, Sigma-Aldrich) was mixed into 10 ml DMSO (ACS grade, Aladdin) solution with stirring at room temperature. 100 μL HCl (37%, Shanghai lingfeng Co. Ltd.) was then added into the turbid mixture before it was transferred into a 250 mL autoclave. The autoclave was then heated to 150 °C in the oven and hold for 6 h (Caution: DMSO is easily exploded at elevated temperature!). It seems the reaction is not air/moisture-sensitive and no special steps were used to remove the air. The obtained dark brown colour product was redispersed in 100 mL anhydrous ethanol with

sonication and washed by centrifugation for 6 times before further characterizations. For the control experiments, 1 g  $\text{NH}_4\text{Cl}$  (99.5%, Aladdin) and 50 mg  $\text{MgB}_2$  (99%, Sigma-Aldrich) was mixed into 10 ml DMSO (ACS grade, Aladdin) solution with stirring at room temperature. 100  $\mu\text{L}$  HCl (37%, Shanghai lingfeng Co. Ltd.) was then added into the turbid mixture and transferred in a 20 mL glass bottle without cap before transferred into the autoclave. The reaction was carried out at 100°C, 120°C, 160°C and 180°C for 6 h. To investigate the formation mechanism, two control experiments were carried out. 1 g  $\text{NH}_4\text{Cl}$  (99.5%, Aladdin) and 50 mg  $\text{MgB}_2$  (99%, Sigma-Aldrich) was mixed into 10 ml DMSO (ACS grade, Aladdin) solution with stirring at room temperature. The mixture was transferred in a 20 mL glass bottle without cap before transferred into the autoclave. The reaction was carried out at 150°C for 6 h. For comparison, 50 mg  $\text{MgB}_2$  (99%, Sigma-Aldrich) was mixed into 10 ml DMSO (ACS grade, Aladdin) solution with stirring at room temperature. 100  $\mu\text{L}$  HCl (37%, Shanghai lingfeng Co. Ltd.) was then added into the mixture and transferred in a 20 mL glass bottle without cap before transferred into the autoclave. The reaction was carried out at 150°C for 6 h. The effect of solvent was studied by changing DMSO to ethanol or DMF for the experiments as follows. 1 g  $\text{NH}_4\text{Cl}$  (99.5%, Aladdin) and 50 mg  $\text{MgB}_2$  (99%, Sigma-Aldrich) was mixed into 10 ml ethanol (ACS grade, Aladdin) solution with stirring at room temperature. 100  $\mu\text{L}$  HCl (37%, Shanghai lingfeng Co. Ltd.) was then added into the turbid mixture and transferred in a 20 mL glass bottle without cap before transferred into the autoclave. The reaction was carried out at 150°C for 6 h. Additionally, 1 g  $\text{NH}_4\text{Cl}$  (99.5%, Aladdin) and 50 mg  $\text{MgB}_2$  (99%, Sigma-Aldrich) was mixed into 10 ml DMF (ACS grade, Aladdin) solution with stirring at room temperature. 100  $\mu\text{L}$  HCl (37%, Shanghai lingfeng Co. Ltd.) was then added into the turbid

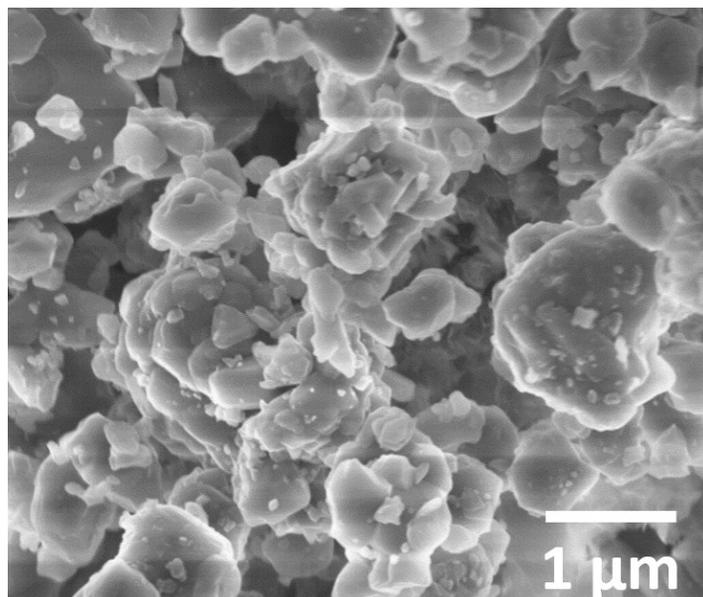
mixture and transferred in a 20 mL glass bottle without cap before transferred into the autoclave. The reaction was carried out at 150°C for 6 h. The obtained product was redispersed in 100 mL anhydrous ethanol with sonication and washed by centrifugation for 6 times before further characterizations.

*Materials characterizations:*

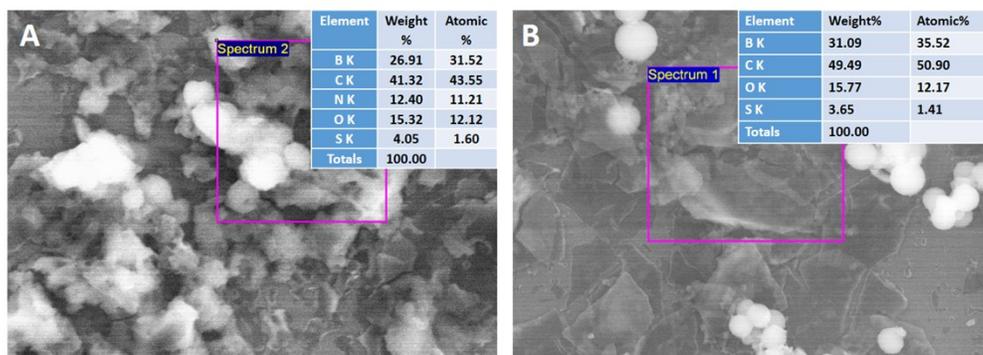
Scanning electron microscopy (SEM, Hitachi S-4800) was used for the morphology analysis. High-resolution transmission electron microscopy (HRTEM, JEOL 2100F) coupled with energy dispersive X-ray spectroscopy (EDS) analyses were performed to gain the crystal structure information. Electron energy loss spectroscopy (EELS) measurements were carried out using a Gatan GIF Quantum 965. Atomic force microscopy (AFM, Bruker Dimension Icon) was used for the plate thickness analysis. XRD (Bruker, D8) was performed with Cu K $\alpha$  radiation ( $\lambda = 1.54 \text{ \AA}$ ) as the X-ray source. The ex situ XRD samples were prepared by disassembling the half-cells after cycling in the glovebox and washed with DMC before covering with paraffin for the characterizations. Fourier Transform Infrared spectroscopy (FTIR) measurement was carried out using Nicolet 6700. The chemical state study of the samples was carried out by X-ray photoelectron spectroscopy (XPS, PHI 5000 VersaProbe) and the binding energies were corrected for specimen charging effects using the C 1s level at 284.6 eV as the reference. Thermogravimetric analysis (TGA, NETZSCH STA409) was conducted with a heating/cooling rate of 10 °C min<sup>-1</sup> between 30 to 600 °C under N<sub>2</sub> atmosphere. B K-edge XANES measurements were performed at the photoemission end station at BL10B beamline of the National Synchrotron Radiation Laboratory (NSRL), China.

*Electrochemical measurements:*

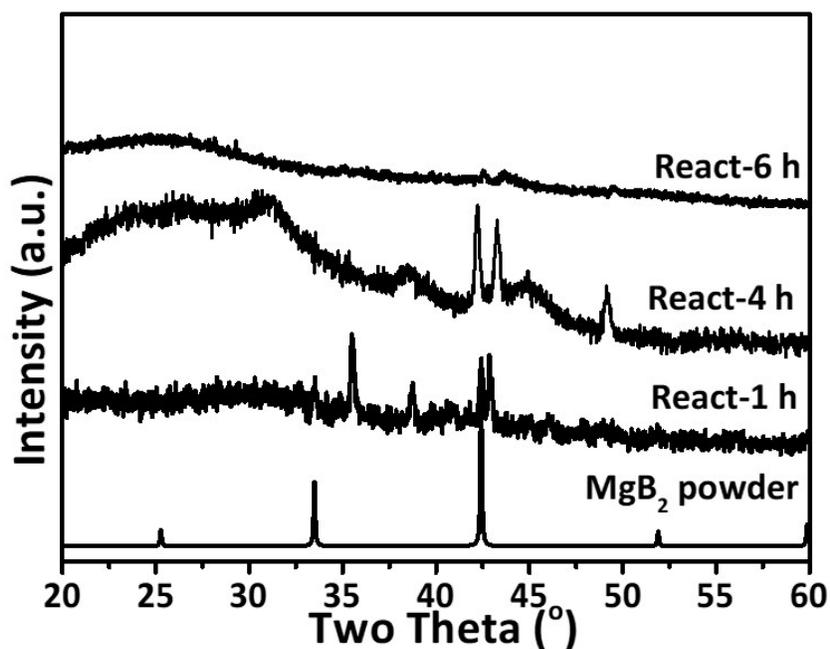
The electrochemical performance of the surface functionalized boron nanoplate was measured with 2032 coin-type half-cells with lithium metal as the anode. The working electrode was prepared by mixing 80% of active materials, 10% of acetylene black and 10% of polyvinylidene fluoride (PVdF) in N-Methyl-2-pyrrolidone (NMP) and the slurry was coated onto a copper foil substrate. 1 mol L<sup>-1</sup> LiPF<sub>6</sub> in a mixture of ethylene carbonate (EC) and DMC (1:1 by volume) was used as electrolyte and Celgard 2300 was chosen as the separator. Cyclic voltammetry (CV) was measured by an electrochemical workstation (CHI 660e) between 0.01-3 V vs. Li/Li<sup>+</sup>. The charge/discharge tests were carried out between 0.01-3 V vs. Li/Li<sup>+</sup> on a Neware CT-4008. For the charge/discharge cycling at different rates, the cells were slowly discharged to 0.01 V at 10 mA g<sup>-1</sup> before the test.



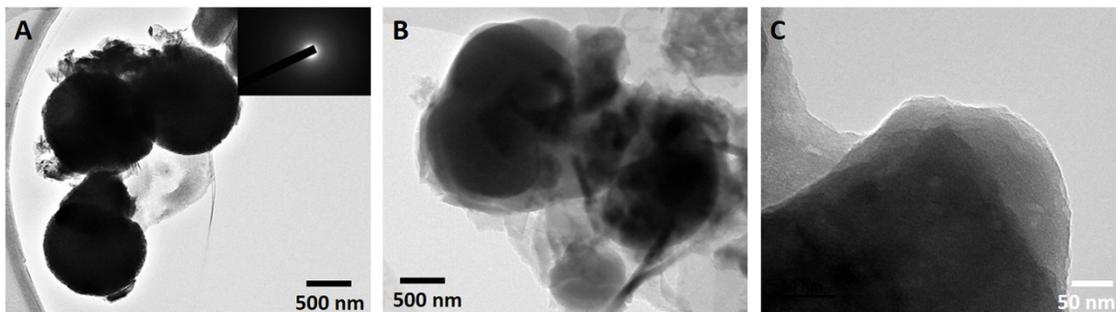
**Figure S1.** SEM image of the pristine MgB<sub>2</sub> particles.



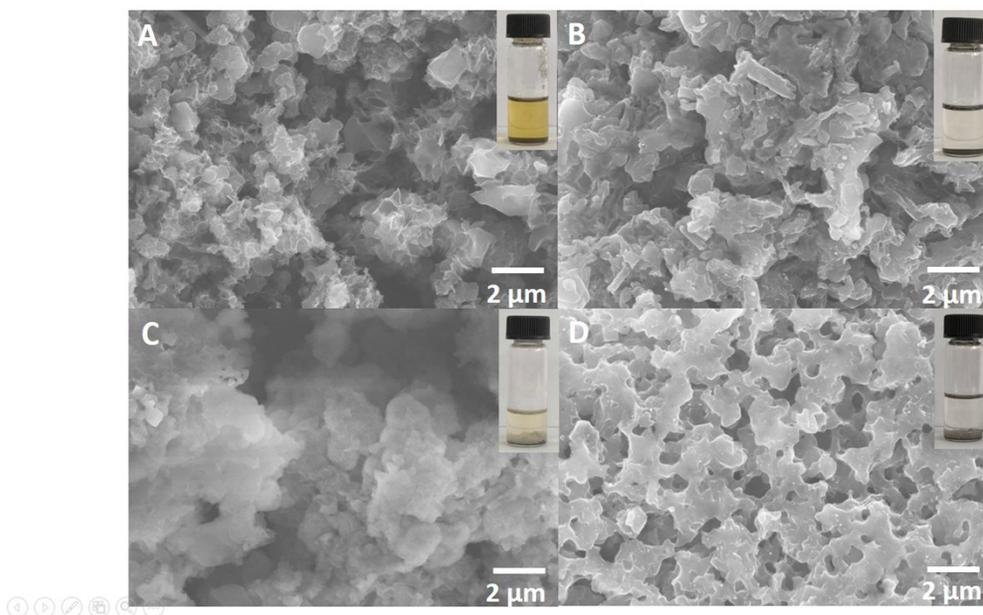
**Figure S2.** EDS of the (A) intermediate products and (B) final obtained boron nanoplates. The elemental N is present at intermediate products and the content of N is negligible in the final boron nanoplates.



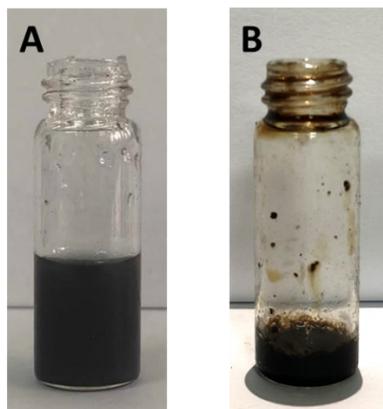
**Figure S3.** XRD pattern of the pristine  $\text{MgB}_2$  particles, the intermediate product obtained after 1 and 4 h reaction and the final product obtained after 6 h reaction. The  $\text{MgB}_2(001)$  plane ( $\sim 25.3^\circ$ ) disappears along with the reaction. The  $\text{MgB}_2(100)$  plane ( $\sim 33.5^\circ$ ) right shifts which possibly results from the buckling of B-layer structure along with the reaction (*Chem*, 2020, 2, 406.). The  $\text{MgB}_2(101)$  plane is split initially and gradually disappears due to the amorphization of the boron layer in the product induced by the intercalation of the  $\text{MgB}_2$  crystal along with the reaction.



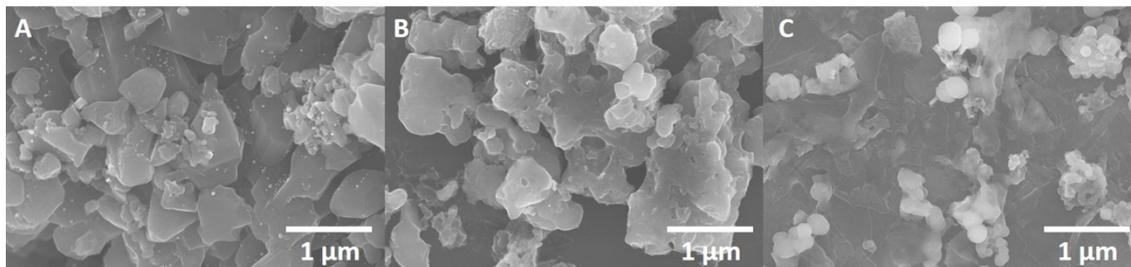
**Figure S4.** TEM images of the intermediate products. (A) the intermediate product obtained after 1h reaction. Inset is the selected area diffracton pattern of the particle which shows the amorphous state of the product. (B) The intermediate product obtained after 4 h reaction, where the exfoliated plates were shown on the surface. (C) The enlarged image of one exfoliated part, which shows the layered structures.



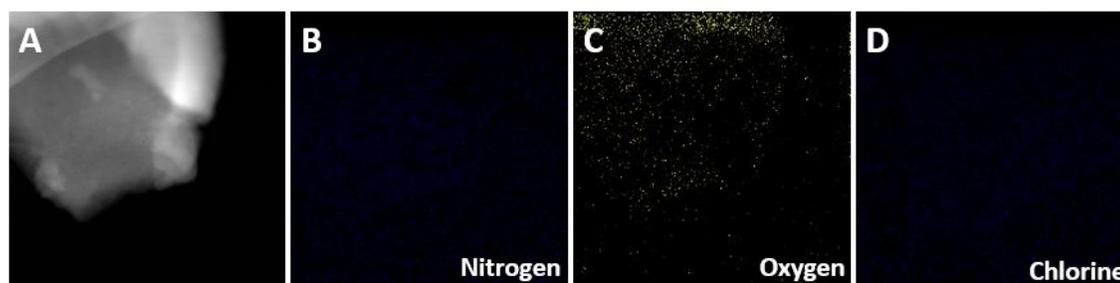
**Figure S5.** SEM images of the reaction products. (A) No HCl was added in the DMSO solution before reaction, (B) No  $\text{NH}_4\text{Cl}$  was added into the DMSO solution before reaction, (C) Ethanol was used as the solvent, (D) DMF was used as the solvent. Insets are the optical images of the glass bottles after reaction.



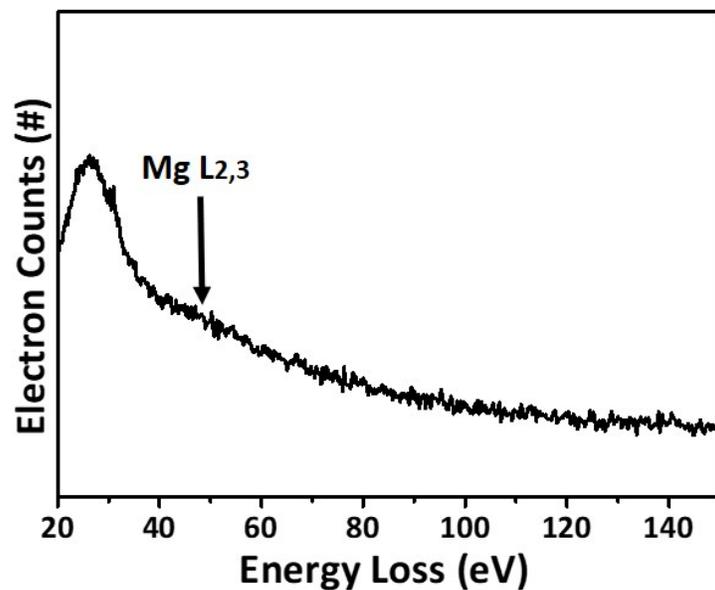
**Figure S6.** Optical image of the (A) mixed precursor and (B) the obtained product after reaction. Only dark-grown colour gel-like product left in the bottle and the remained liquid in the autoclave scattered outside of the bottle.



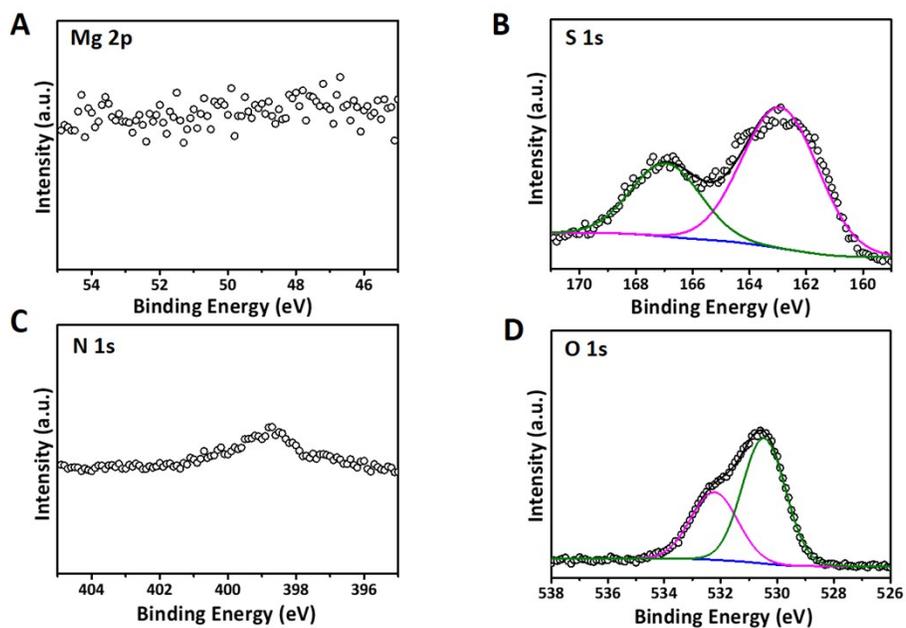
**Figure S7.** SEM images of the products obtained at different reaction temperature. (A) 100 °C , 6 h. (B) 140°C,6 h. (C) 180°C, 6 h.



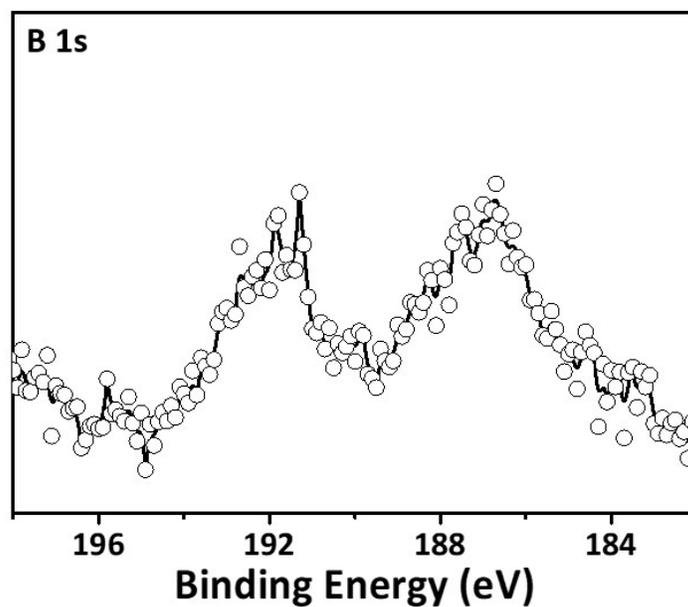
**Figure S8.** (A) HAADF image of one boron nanoplate. (B-D) elemental mapping of the boron nanoplate.



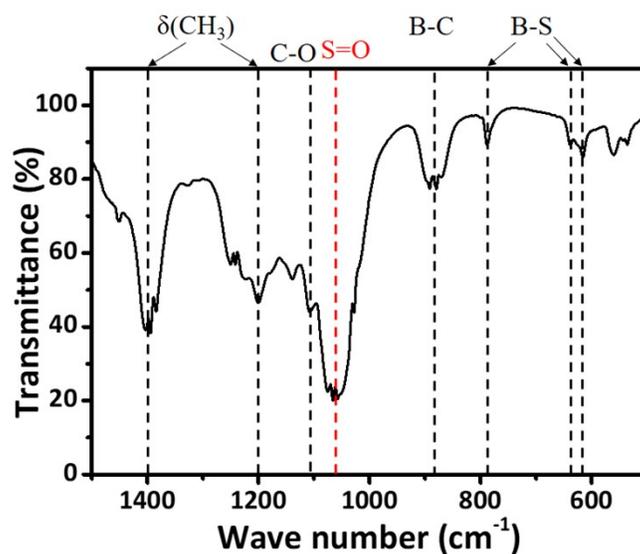
**Figure S9.** EELS spectrum of the boron nanoplates in which no Mg L<sub>2,3</sub> peak was shown.



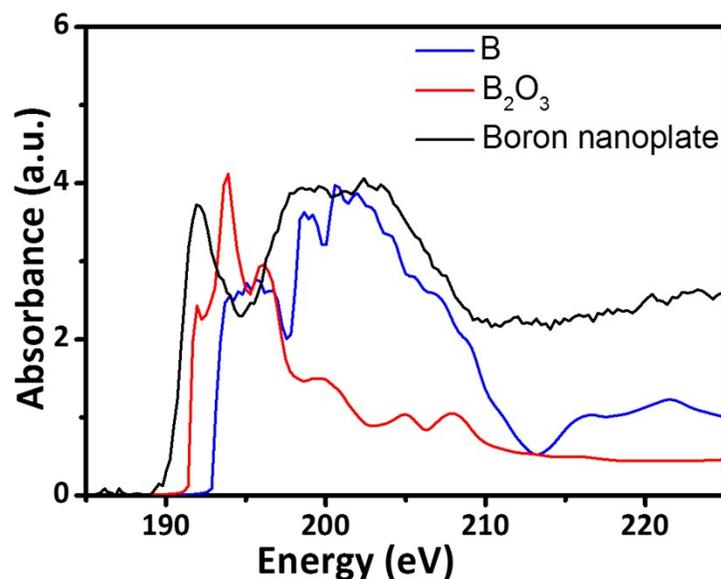
**Figure S10.** XPS (A)Mg 2p, (B) S 1s, (C) N 1s and (D)O 1s of the surface functionalized boron nanoplates.



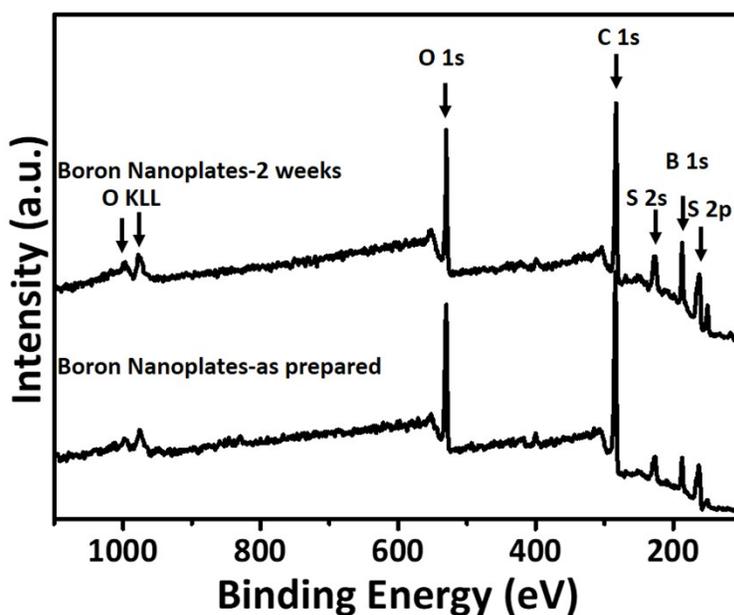
**Figure S11.** XPS B 1s of the MgB<sub>2</sub> particles. The peak ~192 eV results from the surface oxidation of the MgB<sub>2</sub>.



**Figure S12.** The zoom-in fingerprint region of the FTIR spectrum of the boron nanoplates.



**Figure S13.** Boron K-edge XANES of the boron nanoplate, boron and boron (III) oxide. The sharp peak  $\sim 192$  eV in the spectrum is assigned to transition of B 1s electrons to the unoccupied B 2p<sub>z</sub> orbital and the broad peak between 195 to 210 eV may be resulted from a multiple-scattering resonance. Therefore, the B in the boron nanoplates presents distinct chemical states compared with it in the bulk boron or boron oxide.



**Figure S14.** XPS survey spectra of the as prepared boron nanoplates and the nanoplates exposed in air for two weeks. The estimated oxygen content varied from 6.1 at.% (as prepared sample) to 7.3 at.% (exposed sample). The almost unchanged oxygen content implies the high environmental stability of our synthesized boron nanoplates.

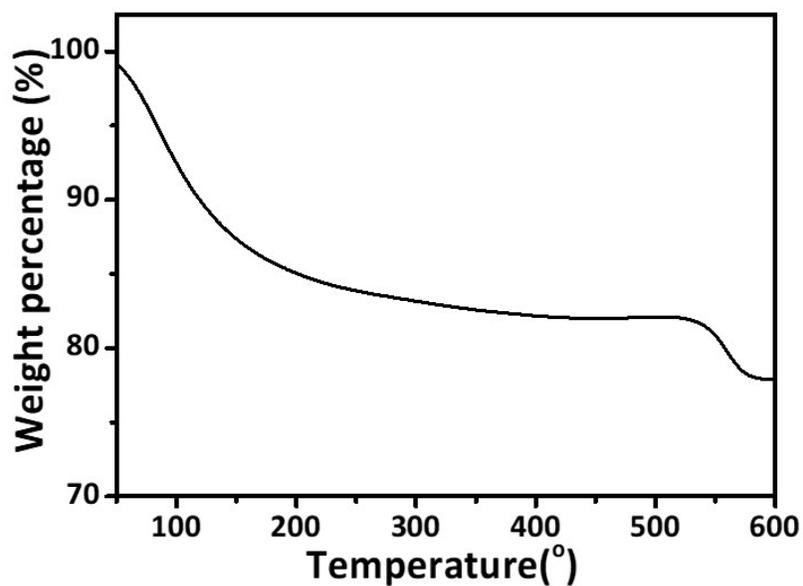


Figure S15. TGA of the surface functionalized boron nanoplates.

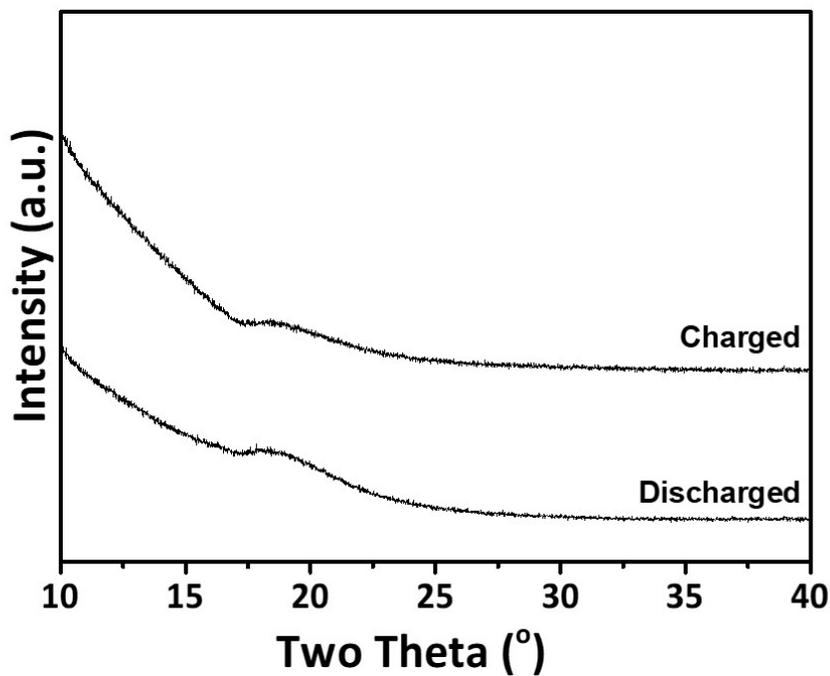


Figure S16. Ex situ XRD patterns of the electrodes charged after 100 cycle and discharged after 100 cycle at  $0.5 \text{ A g}^{-1}$ , respectively. No obvious differences were shown on the patterns.

**Table S1.** Elemental composition of the surface functionalized boron nanoplates

	N (wt.%)	C (wt.%)	H (wt%)	S (wt.%)	O (wt.%)	Cl (wt.%)
Boron nanoplate	0.16	31.77	4.847	20.892	1.54	0.61