

Supplementary Information

Graphene reduced from magnesiothermic reaction†

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Experimental

Preparation of Graphite Oxide (GO). GO was prepared using a modified Hummer methods.¹⁹ Typically, 4 g of natural graphite flakes (> 100 mesh, Aldrich) was put into an mixture of H₂SO₄ (15 mL, 98 wt%), K₂S₂O₈ (5 g), and P₂O₅ (5 g) at 80 °C. The resultant product was collected after a cooling procedure for 12 hrs following a careful washing with deionized water until the pH value of the rinse water rises close to 7. The preoxidized graphite powder (1 g) and 0.75 g of NaNO₃ were placed in a flask followed by adding H₂SO₄ (75 mL, 98 wt%) under stirring in an ice-water bath. Then, 3 g of KMnO₄ was slowly added to the graphite/NaNO₃/H₂SO₄ mixture over 90 min. After the mixture was stirred at ~ 35 °C for 6 hrs and stayed overnight at room temperature, 3 mL of H₂O₂ (30 wt%) and 300 mL H₂O was added, when the solution color turned bright yellow. The mixture was filtered and washed with an aqueous HCl solution and a large amount of deionized water. Finally, GO film was collected after dried at 80 °C overnight.

Preparation of G-Mg. GO-MW was first prepared as described before (see ref. 14a in main text). GO film was treated in a commercial microwave oven (Emerson MW 8995B) at ambient conditions at a power of 720 W for 1 min. Then, GO-MW and Mg powder (1:1 in weight) was ground together under an Ar atmosphere and sealed in a Swagelok reactor. Then, the GO-MW/Mg mixture was heated in a tube furnace at 650°C for 2 hrs under an Ar atmosphere. Finally, the graphene product, referred to as G-Mg, was collected after being washed with an aqueous solution of HCl, and vacuum-dried at 60°C overnight.

Characterization. X-Ray diffraction (XRD) patterns were collected using a Rigaku Ultima IV Diffractometer with Cu K α irradiation ($\lambda = 1.5406 \text{ \AA}$). WITec confocal Raman with a 514 nm laser source was used to collect the Raman spectra. For ease of comparison, the XRD and Raman results are normalized to the intensity of the strongest peak. The morphology was examined by field emission scanning electron microscopy (FESEM) using an FEI NOVA 230 high resolution SEM. Transmission electron microscopy (TEM) images were collected by a FEI Titan 80-200. Nitrogen sorption measurements were performed on a Micromeritics TriStar II 3020 analyzer at 77 K. X-ray Photoelectron Spectroscopy (XPS) measurements were performed in a Physical Electrons Quantera Scanning ESCA Microprobe with a focused monochromatic Al K α X-ray (1486.6 eV) source for excitation. The X-ray beam used was a 25 W, 100 μm X-ray beam spot at the sample. The binding energy (BE) scale was calibrated using the Cu 2p $_{3/2}$ feature at $932.62 \pm 0.05 \text{ eV}$ and Au 4f at $83.96 \pm 0.05 \text{ eV}$. The ion gun used in this system was a standard Quantera ion gun, and the sputter depth profiles were acquired using a 1 KeV argon-ion beam rastered over a 3 mm x 3 mm area. To minimize charging artifacts, the XPS data were collected with 1 eV, 20 μA electrons and low-energy Ar $^+$ ions.

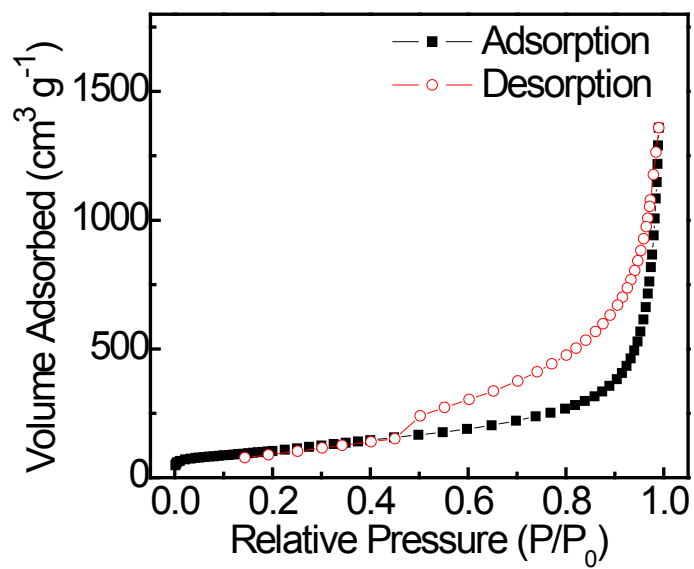


Fig. S1 N₂ adsorption/desorption isotherm of GO-MW.