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Supporting Information

Insight into the Electrochemical Activation of Carbon-Based Cathodes for Hydrogen Evolution Reaction

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1. Loading of Pt/C and carbon nanohorns on glassy carbon electrode

For the purpose of comparison on the catalytic performance for HER, the commercial Pt/C powder (20 w% Pt on Vulcan XC-72) was also measured under the similar conditions by loading it on a glassy carbon working electrode (GCE) with drop casting method. A glassy carbon disk electrode with a diameter of 5 mm was used. Before loading, the GCE surface was polished with the slurry of α-Al_2O_3 powder with different particle sizes (500 nm, 100 nm, and 50 nm), then ultrasonicated with DI water, ethanol, and DI water accordingly for 5 minutes and dried with pure nitrogen gas. Next, 2.0 mg Pt/C powder was ultrasonically dispersed in 0.5 mL Isopropyl alcohol and 50.0 μL 0.1% Nafion solution. Then, 30 μL of the above-mentioned catalyst dispersion was transferred onto the clean glassy carbon electrode and dried in the open air. The loading density of Pt/C powder is ~0.61 mg · cm^{-2}. The same quantity of CNHs was also loaded on the GCE with the same loading procedure for Pt/C and the catalytic activity of CNHs for HER was also determined by the same procedure exploited on graphite rod.

2. EDS of the Pt wire

Before the EDS determination, the Pt wire was first polished with SiC abrasive paper (Eagle, 1500 type) and then cleaned with water, ethanol alcohol and water accordingly under the ultrasonication. A piece of Pt wire with a length of about 3 mm was used for analysis and the voltage applied for the measurement was 18 kV. The EDS pattern of the commercial Pt wire is then shown in Figure S1.

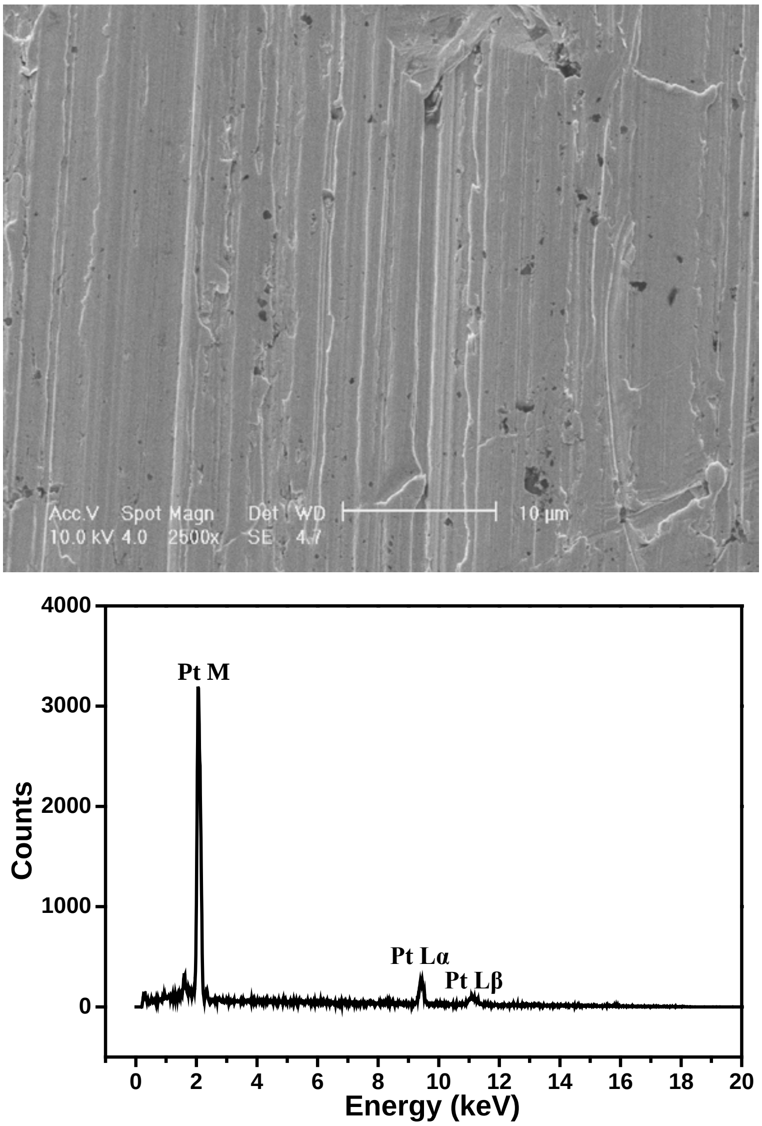

Figure S1. SEM image (top) and the corresponding EDS spectra (bottom) of the commercial Pt wire.

3. EDS characterization of the initial graphite electrode

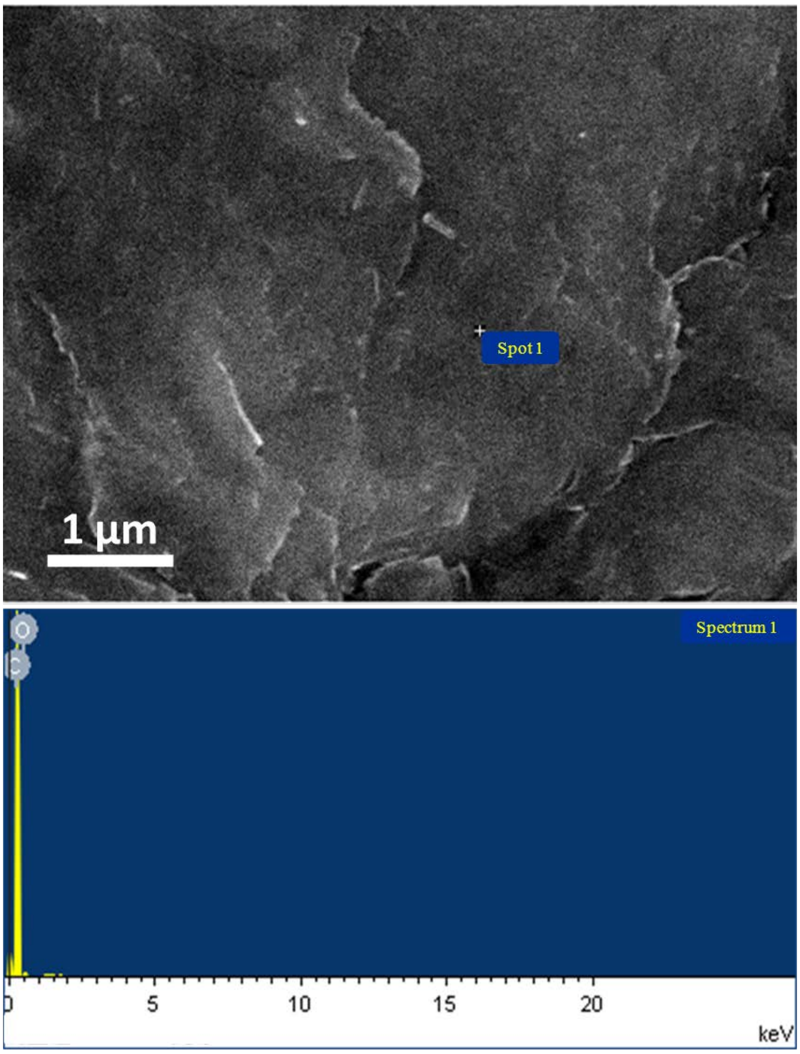

Figure S2. SEM image (top) and the corresponding EDS spectra (bottom) of the initial graphite electrode surface.

4. EDS characterization of the Pt-GE activated with Pt wire

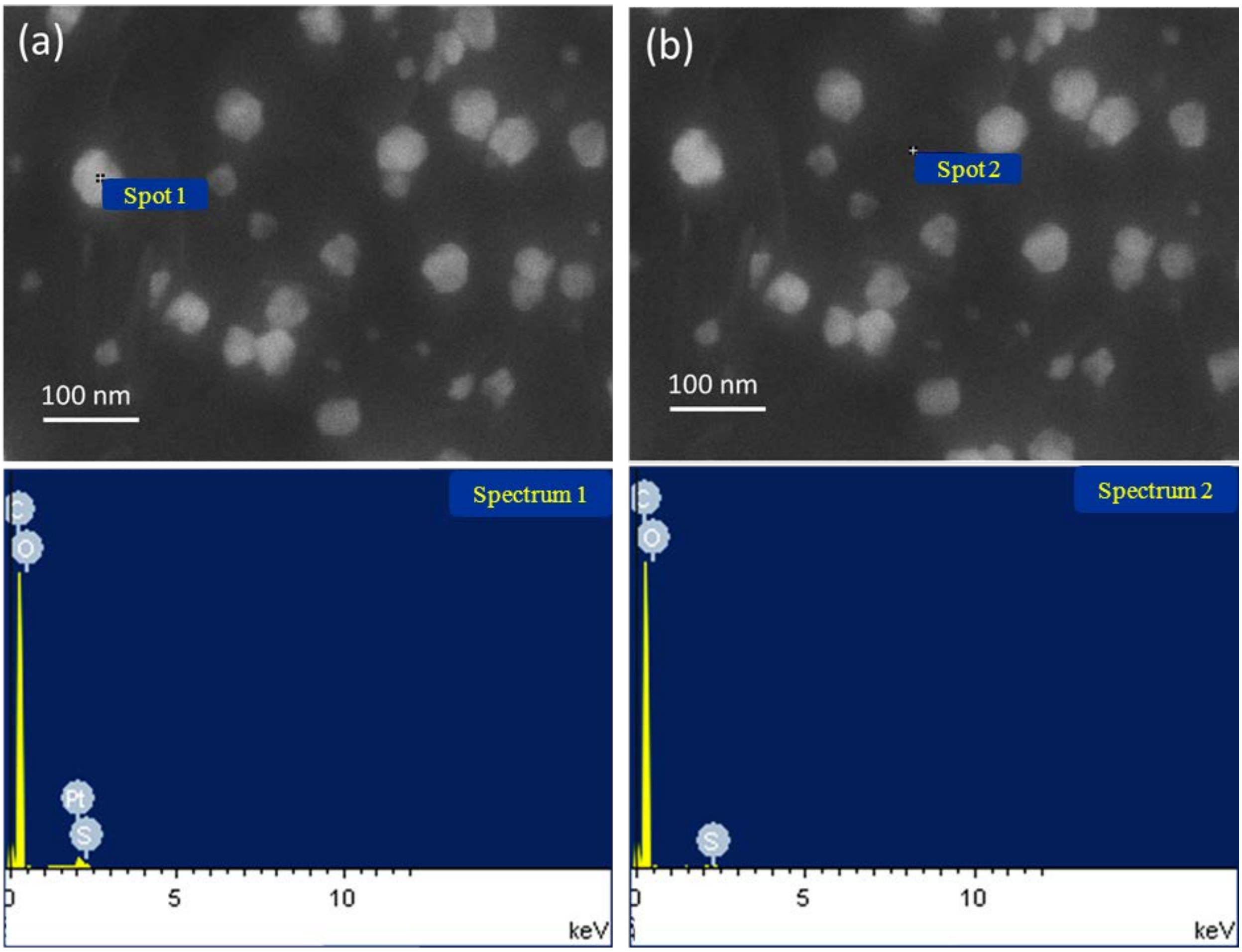

Figure S3. EDS spectra collected on different surface location of Pt-GE activated with Pt wire as the counter electrode, (a) on nanoparticle; (b) on the graphite layer.

5. HER performance of graphite electrodes after different cycles of activation with different counter electrodes

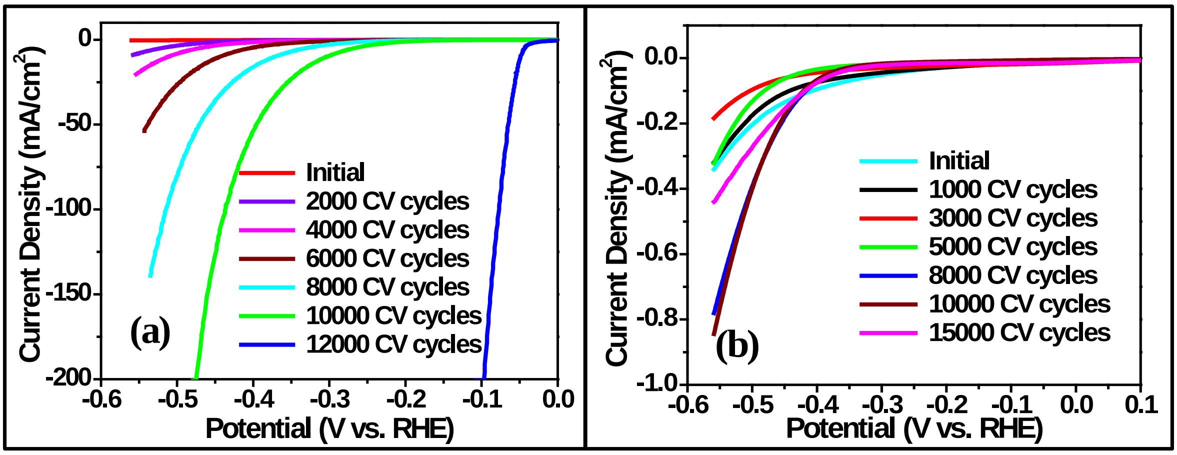

Figure S4. LSV curves on HER of graphite electrodes after different cycles of activation when using Pt wire (a) or graphite rod (b) as counter electrode, respectively.

6. The morphology change of Pt-GE after different cycles of activation with Pt wire as the counter electrodes

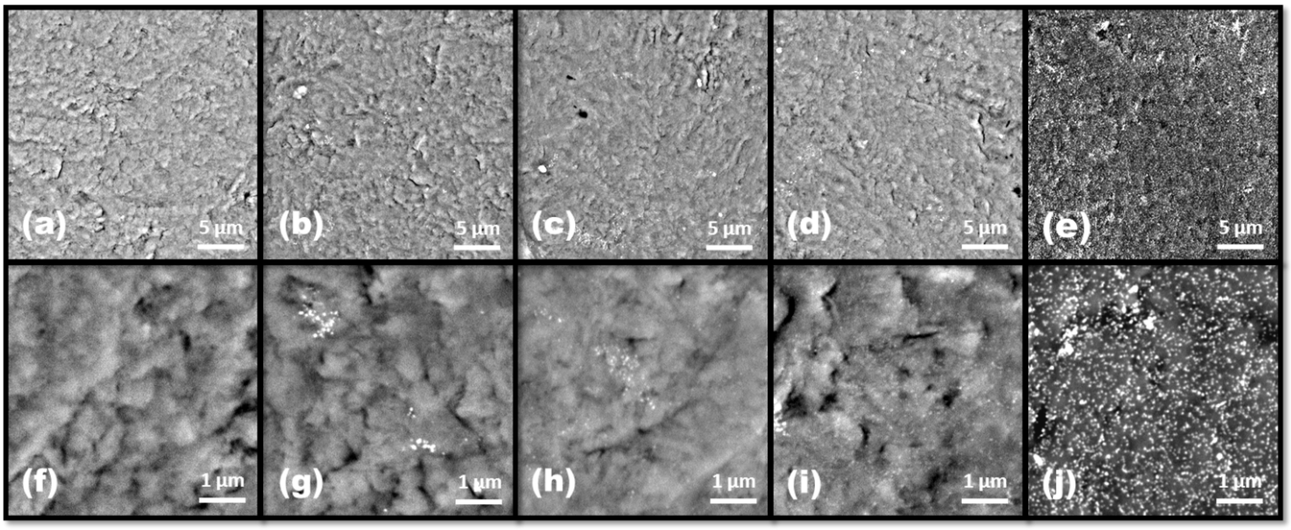

Figure S5. SEM images of the graphite electrode surface after different CV cycles when using a Pt wire as counter electrode. (a, f) 0 cycle, (b, g) 4000 cycles, (c, h) 8000 cycles, (d, i) 10000 cycles, (e, j) 12000 cycles, respectively.

7. TEM characterization of carbon nanohorns

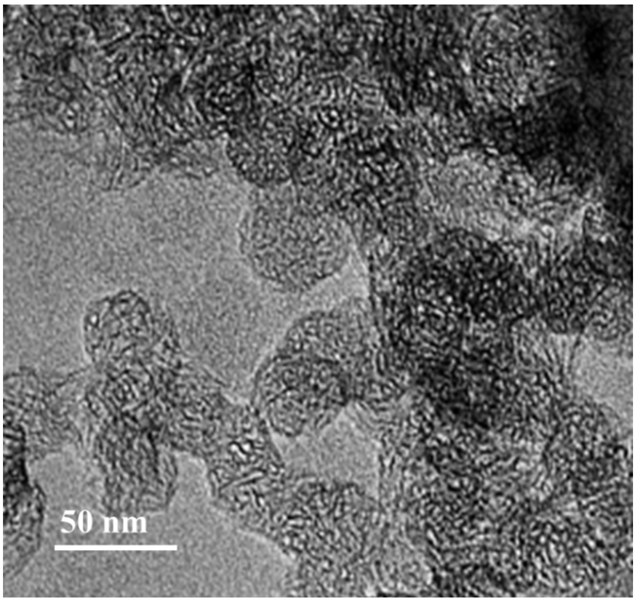

Figure S6. The TEM picture of CNHs.