## **Supporting Information**

## Electrodeposition of sulfur-engineered amorphous nickel hydroxides on MIL-53(Fe) nanosheets to accelerate the Oxygen Evolution Reaction

Fang Wu,<sup>‡</sup> Xiaoxue Guo,<sup>‡</sup> Gazi Hao, Yubing Hu<sup>\*</sup> and Wei Jiang<sup>\*</sup>

School of Chemical Engineering, Nanjing University of Science and Technology, Nanjing 210094, China.

E-mail: <a href="mailto:superfine\_jw@126.com">superfine\_jw@126.com</a>, <a href="https://www.hyb.avanta.com">hyb@njust.edu.cn</a>



Figure S1. The SEM image of Ni foam.



Figure S2. TEM spectra of (a) MIL-53(Fe) and (b) Ni/MIL-53(Fe).



Figure S3. FT-IR spectrums of MIL-53(Fe), Ni-S/MIL-53(Fe) and Ni/MIL-53(Fe).



Figure S4. Raman spectrums of Ni/NF, Ni-S/NF, Ni/MIL-53(Fe) and Ni-S/MIL-53(Fe).



Figure S5. XPS spectra of Fe 2p for MIL-53(Fe) and Ni-S/MIL-53(Fe).



Figure S6. High resolution XPS spectra for the (a) Ni 2p, (b) Fe 2p, (c) O 1s and (d) S 2p regions of Ni-S/MIL-53(Fe) before and after 40 h of stability test.



Figure S7. Polarization curves of Ni-S/MIL-53(Fe) with various deposition time in 1 M KOH.



**Figure S8.** SEM-EDX spectra of Ni-S/MIL-53(Fe) with different electrodeposition time of (a) 2, (b) 5, (c) 15, (d) 25, (e) 40 and (f) 50 min, respectively.



Figure S9. (a)  $N_2$  adsorption/desorption isotherms, and (b) pore size distribution curve of MIL-53(Fe) electrode.



Figure S10. (a) N<sub>2</sub> adsorption/desorption isotherms, and (b) pore size distribution curve of Ni/MIL-53(Fe) electrode.



Figure S11. (a)  $N_2$  adsorption/desorption isotherms, and (b) pore size distribution curve of Ni-S/MIL-53(Fe) electrode.



**Figure S12.** CVs of (a) Ni-S/MIL-53(Fe), (b)Ni/MIL-53(Fe), (c) MIL-53(Fe), (d) Ni-S/NF and (e) Ni/NF in the region of -0.05-0.05 V vs Hg/HgO at scan rates of 40, 60, 80, 100 and 120 mV s<sup>-1</sup>.



**Figure S13.** The OER activity of MIL-53(Fe), Ni/MIL-53(Fe) and Ni-S/MIL-53(Fe) after the electrochemical surface area normalization.



Figure S14. (a) XRD pattern and (b) SEM image of Ni-S/MIL-53(Fe) after 40 h of stability test.



Figure S15. Roman spectra of Ni-S/MIL-53(Fe) before and after 40 h of stability test.