Synthesis of Ni_3S_2 with various sulphur sources and their HER and OER performance

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Physical characterization

The XRD pattern of the catalyst was recorded at 2θ scan rate of 5 ° min^{-1} (scan

from 5 ° and 90 °) on Shimadzu XD-3A (Japan) using filtered Cu-Kα radiation (λ = 0.15418nm) generated at 40 kV and 30 mA. The microstructure was investigated by Carl Zeiss scanning electron microscopy (SEM). X-ray photoelectron spectra (XPS) was acquired with a VG Escalab210 spectrometer fitted with Mg 300 W X-ray source. Transmission electron micrographs (TEM),high angle annular dark field scanning transmission electronmicroscopy (STEM) images and selected area electron diffraction (SAED) patterns of the catalysts were obtained using a JEOL (JEM-2000 FX) microscope operating at 200 kV. Electrochemical impedance spectrum was measured from 0.01 to 1,000,000 Hz for the HER and the OER.

Electrochemical measurements

The electrochemical performance of as-prepared samples were all evaluated using CHI 660E electrochemical workstation. The electrochemical analysis was carried out in three-electrode configuration with as-prepared samples (with the area 1×1 cm^2), active carbon (AC), Hg/HgO (1.0 M KOH) as working electrode, counter electrode and reference electrode respectively. Linear sweep voltammetry (LSV) and cyclic voltammetry (CV) were carried out in aqueous KOH electrolyte (1.0 M). at corresponding potentials. All the obtained potentials in this work were converted to the potential versus the reversible hydrogen electrode (RHE) via the equation: $E_{RHE} = E_{Hg/HgO} + 0.059\ pH + 0.14\ V$. Electrochemical double-layer capacitance (EDLC) was employed to calculate the electrochemical surface area (ECSA) via cyclic voltammetry (CV) analysis at various scan rates. *iR* compensation (90%) was used for all the electrochemical experiments.

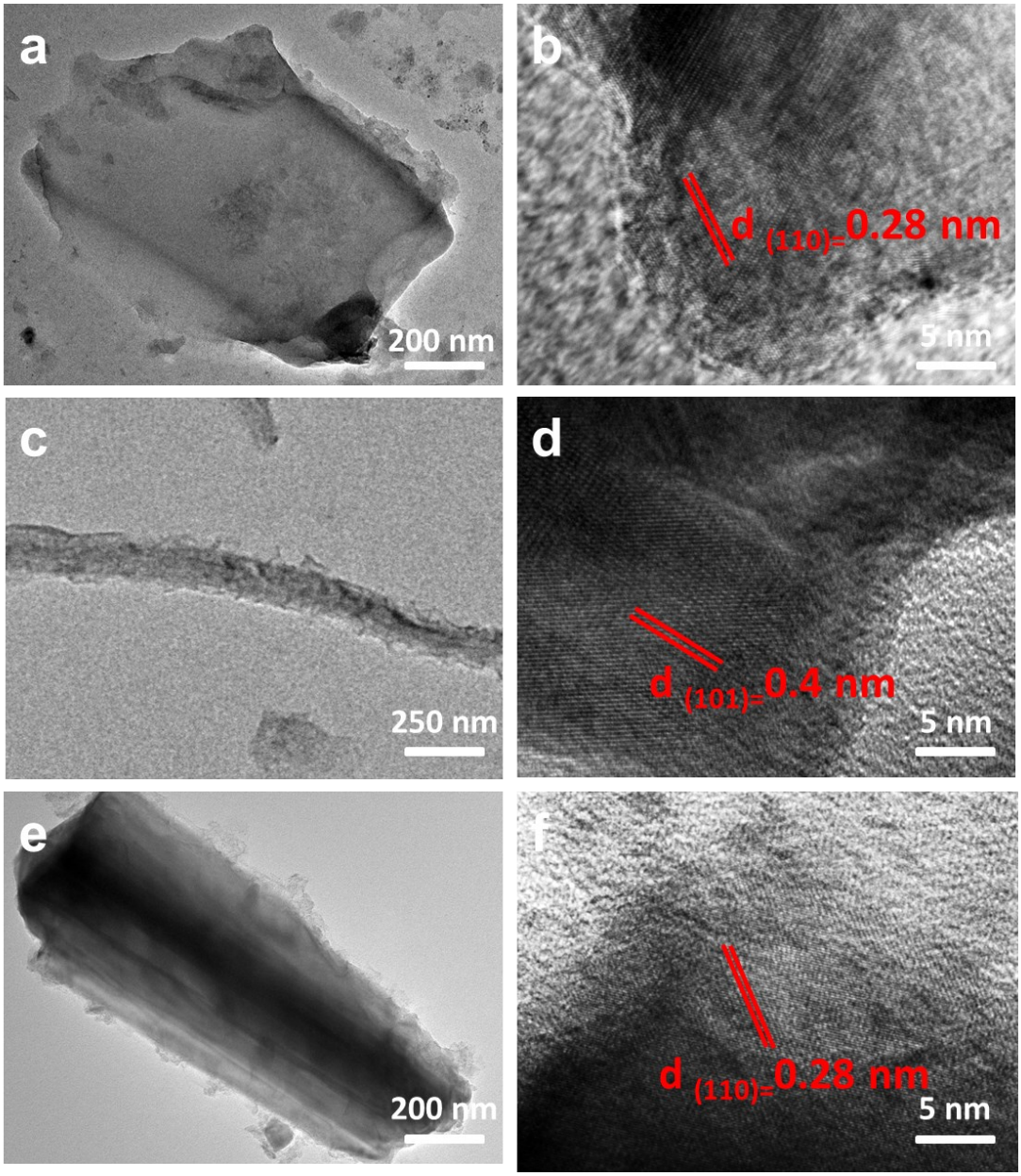

Figure S1. TEM images of Ni_3S_2-1 (a,b); Ni_3S_2-2 (c,d); Ni_3S_2-3 (e,f).

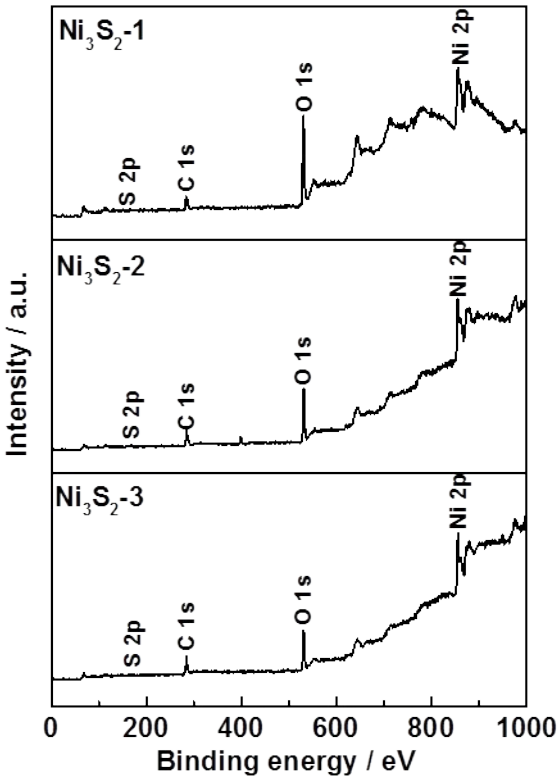

Figure S2. Survey XPS spectrum of Ni_3S_2-1, Ni_3S_2-2, Ni_3S_2-3.

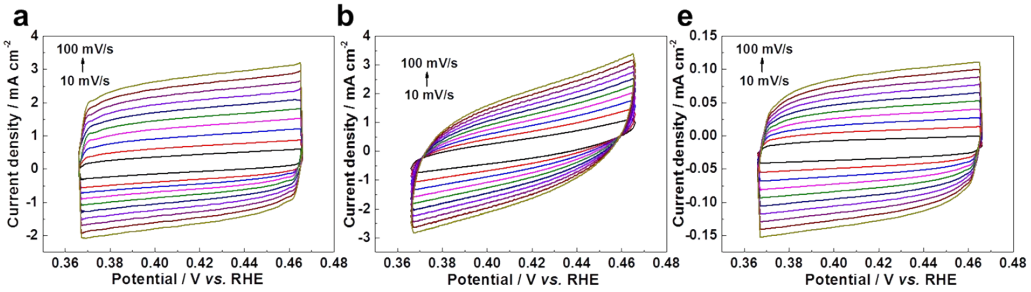

Figure S3. HER CV curves for Ni_3S_2-1; (a)Ni_3S_2-2 (b); and Ni_3S_2-3 (c).

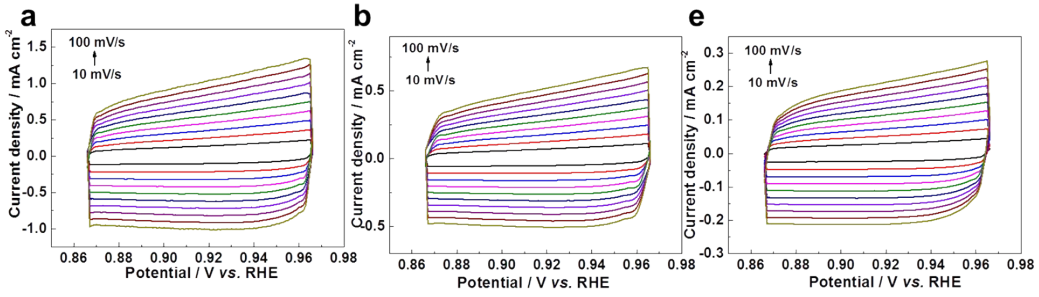

Figure S4. OER CV curves for Ni_3S_2-1; (a)Ni_3S_2-2 (b); and Ni_3S_2-3 (c).

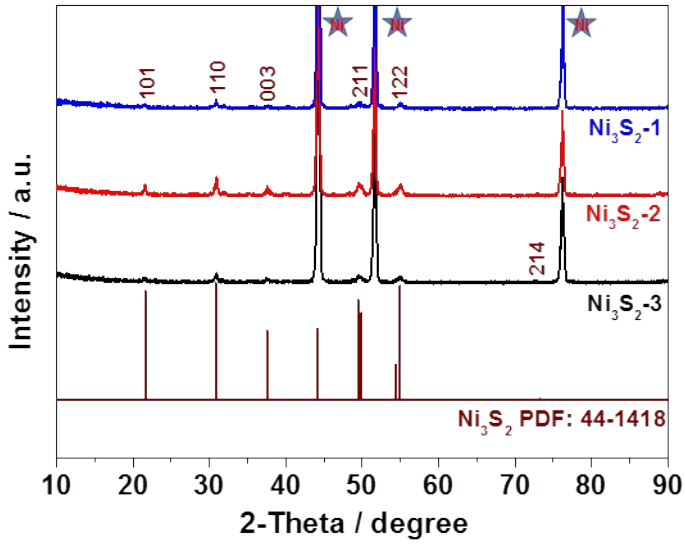

Figure S5. XRD patterns of Ni_3S_2-1, Ni_3S_2-2 and Ni_3S_2-3 after stability test.

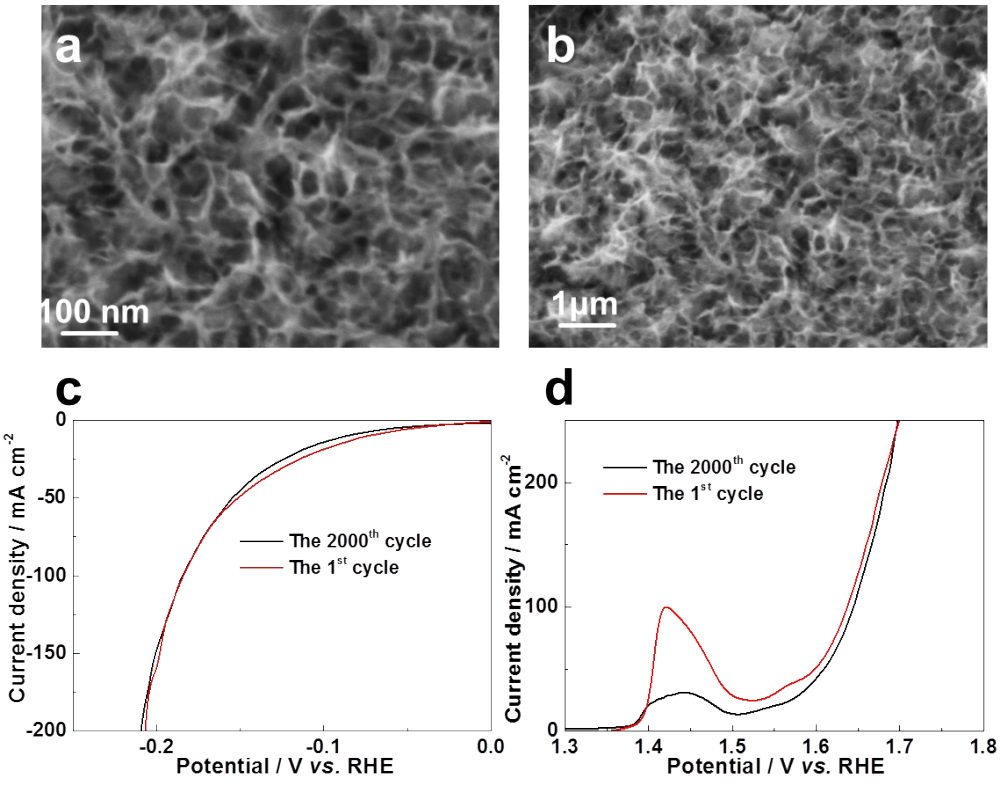

Figure S6. SEM image of Ni_3S_2-1 after HER and OER measurement (a,b); HER(c) and OER(d) LSV curves of the 1st and 2,000th cycles.

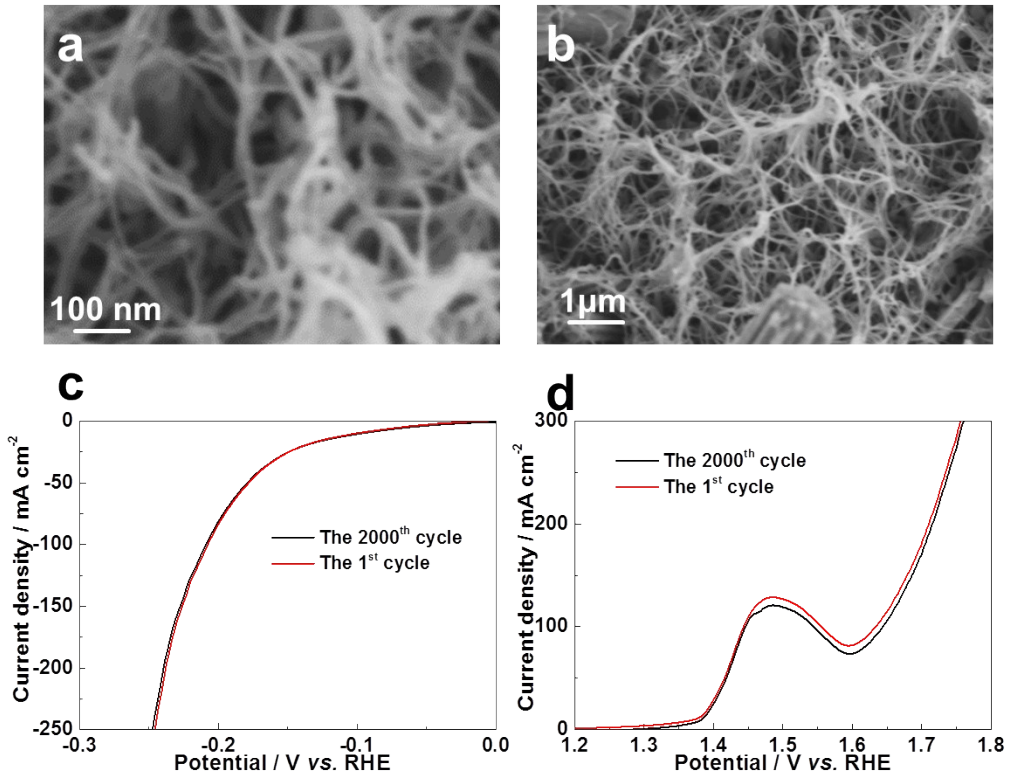

Figure S7. SEM image of Ni_3S_2-2 after HER and OER measurement (a,b); HER(c) and OER(d) LSV curves of the 1st and 2,000th cycles.

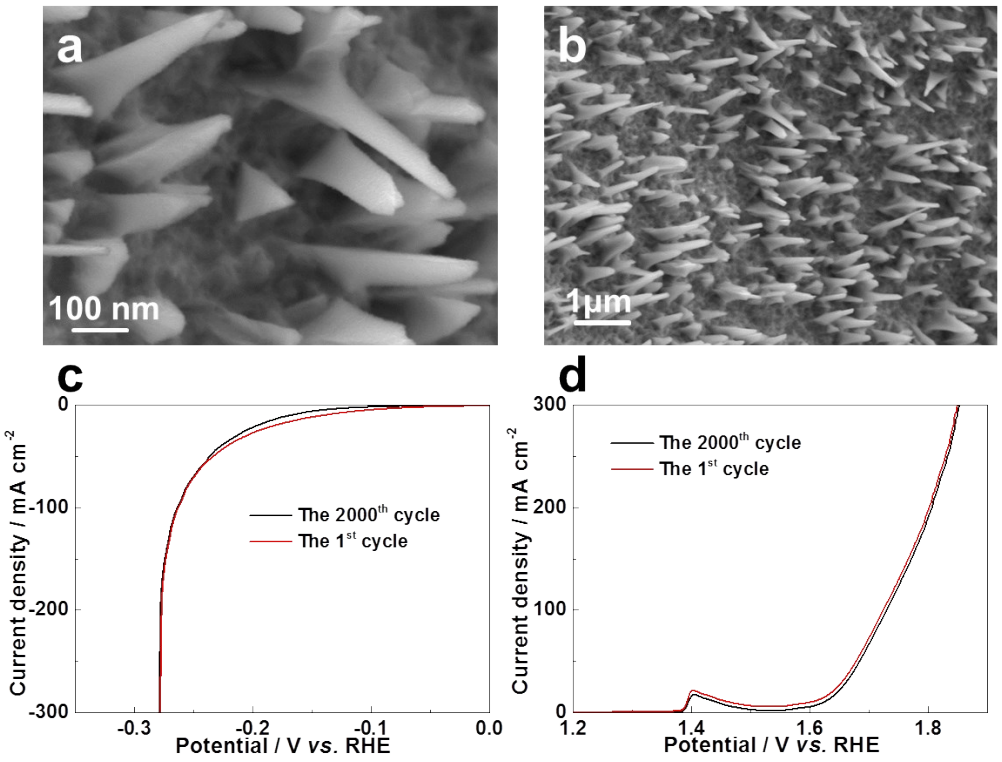

Figure S8. SEM image of Ni_3S_2-3 after HER and OER measurement (a,b); HER(c) and OER(d) LSV curves of the 1st and 2,000th cycles.

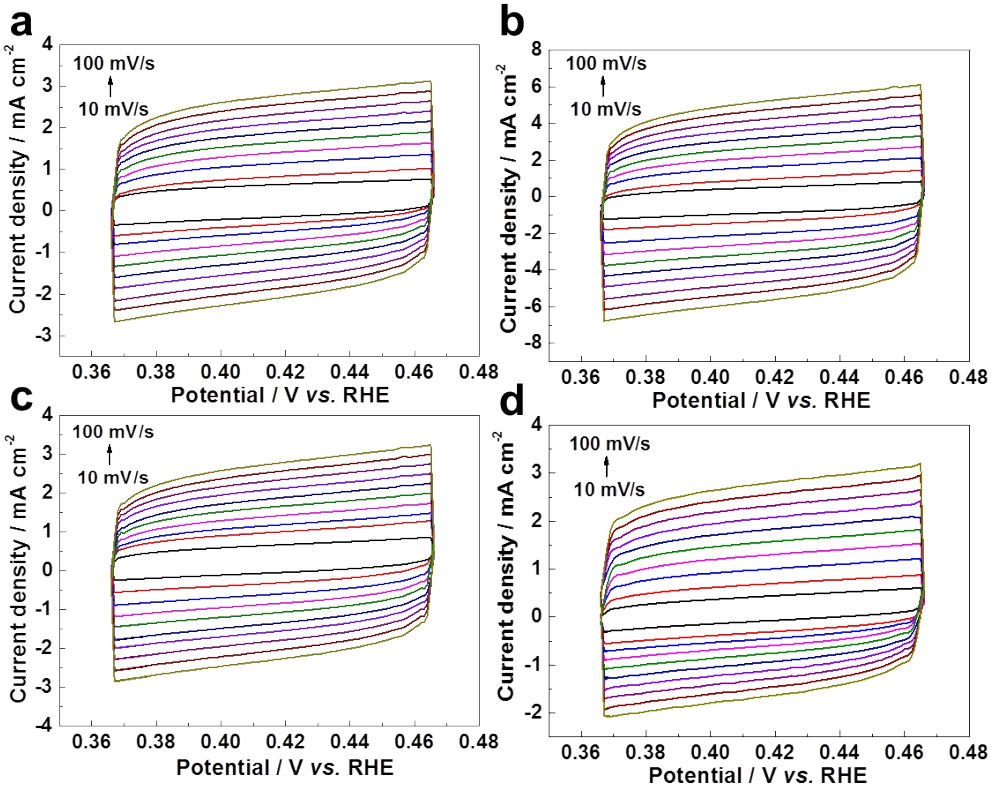

Figure S9. HER CV curves for Ni_3S_2-1-100; (a) Ni_3S_2-2-120 (b); Ni_3S_2-3-140 (c); Ni_3S_2-3-160 (d).

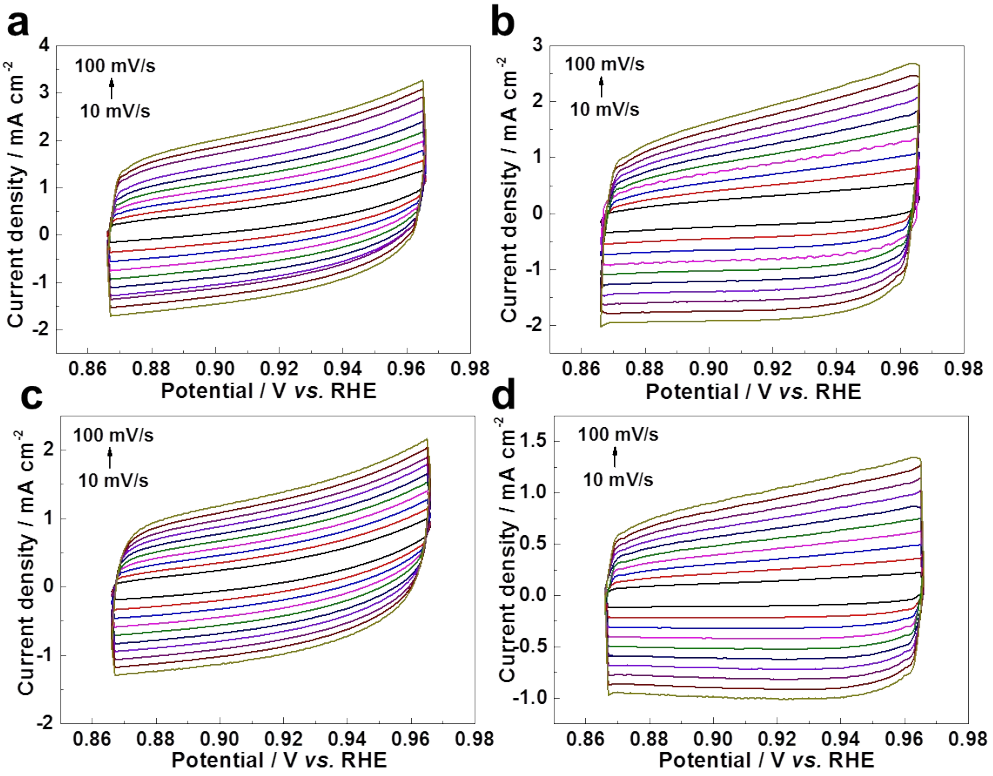

Figure S10. OER CV curves for Ni_3S_2-1-100 (a); Ni_3S_2-2-120 (b); Ni_3S_2-3-140 (c); Ni_3S_2-3-160 (d).

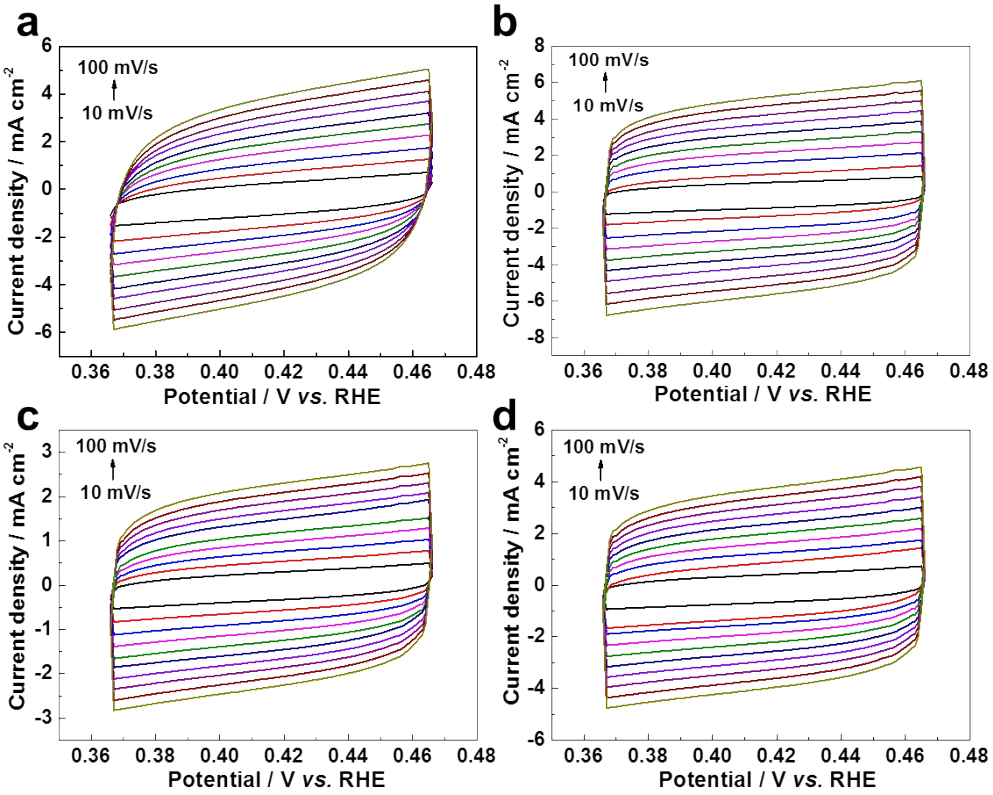

Figure S11. HER CV curves for Ni_3S_2-1-100-2 (a); Ni_3S_2-2-120-4 (b); Ni_3S_2-3-140-6 (c); Ni_3S_2-3-160-8 (d).

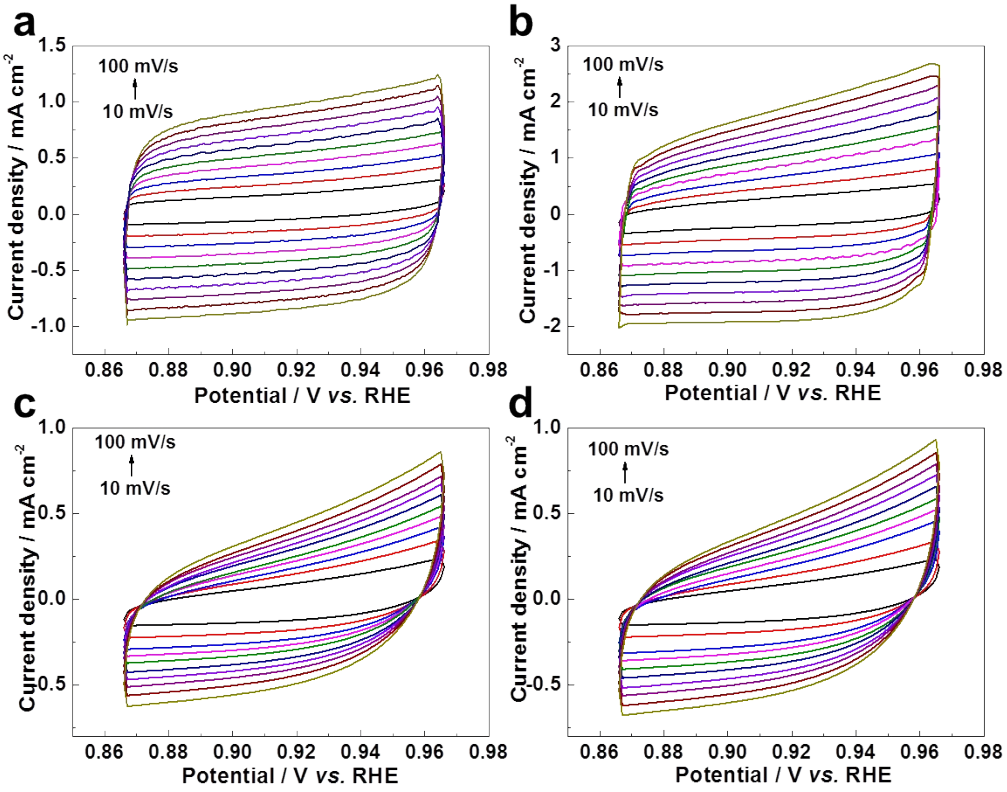

Figure S12. OER CV curves for Ni_3S_2-1-100-2 (a); Ni_3S_2-2-120-4 (b); Ni_3S_2-3-140-6 (c); Ni_3S_2-3-160-8 (d).