

Supporting information for:

Sulfur vacancies modulated nickel-doped Co_4S_3 hollow nanocubes/nitrogen-doped V_2CT_x MXene nanosheets composite for optimizing hydrogen evolution reaction

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Characterization

X-ray diffraction (XRD) measurements were implemented by a powder X-ray diffraction system (Rigaku, TTR-III). The X-ray photoelectron spectroscopy (XPS) measurements were implemented by a Thermo ESCALAB 250Xi spectrometer with monochromatic Al K α radiation ($h\nu=1486.6$ eV). The structure was investigated by scanning electron microscope (FE-SEM) (Hitachi, SU8000) and a transmission electron microscopy (TEM) (JEOL, JEM-2010, 200 kV). Nitrogen adsorption-desorption experiments were carried out at 77.35 K by means of an Autosorb-1 (Quantachrome Instruments) analyzer.

Electrochemical measurements

The measurements on the electrochemical performances were performed using a CHI 660E electrochemical workstation in a standard three-electrode mode. All electrochemical performance tests were performed in 0.5 M H₂SO₄ solution. The working electrode was prepared by passing the slurry containing 4 mg Ni-Co₄S₃ (Sv)/N-V₂CT_x (85 wt.%) with acetyleneblack (10 wt.%) and PTFE (5 wt.%) in 10 mL absolute ethanol. After that, the electrode was made by heating at 60 °C for 40 min. The as-synthesized Ni-Co₄S₃ (Sv)/N-V₂CT_x slurry was coated onto the glass carbon electrode by applying suitable compressive force. The working electrode, reference electrode, and counter electrode are self-supporting electrode carrying electrocatalysts, saturated calomel electrode and graphite rods, respectively. The HER potentials used in this work are corrected with a reversible hydrogen electrode.

Table S1 The various porous parameters of the samples

Samples	Specific Surface Area	Pore Diameter
V_2CT_x	6.65 m ² /g	3.39 nm
Ni-Co ₄ S ₃ (S _v)/N- V_2CT_x	12.58 m ² /g	2.20 nm

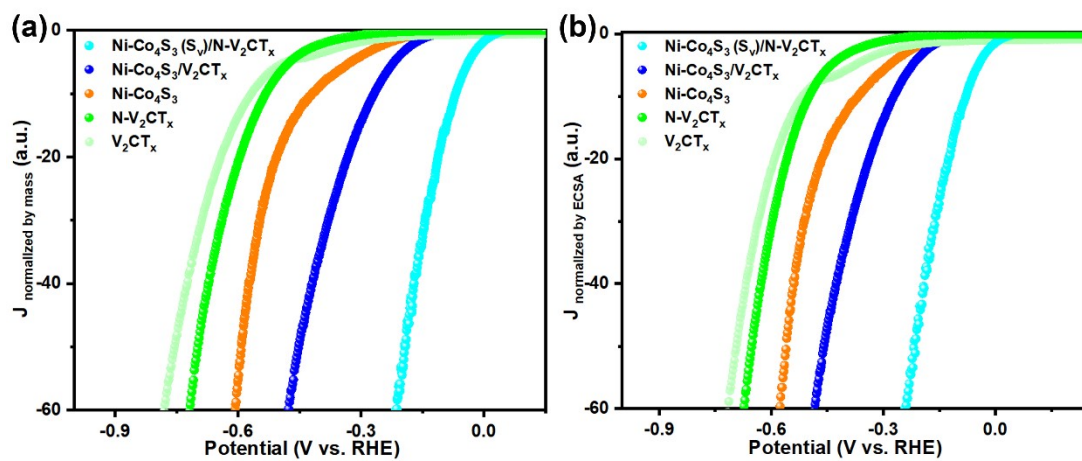


Figure S1 LSV curves of catalysts after normalizing the current by loading mass (a) electrochemical active surface area (b).

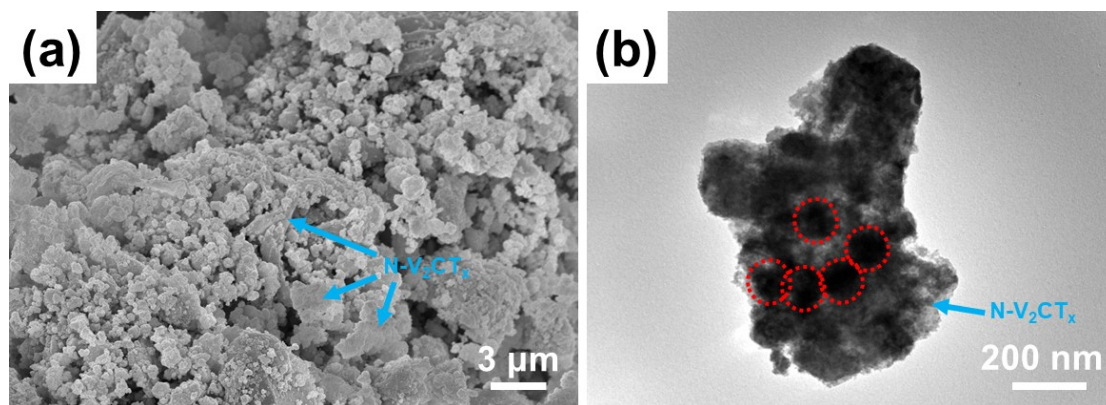


Figure S2 SEM (a) and TEM (b) images of $\text{Ni-Co}_4\text{S}_3$ (S_v)/ $\text{N-V}_2\text{CT}_x$ after test.