Supplementary Information (SI) for Catalysis Science & Technology.

Supporting Information

Construction of Ni_3S_4-CeO_2 nanocomposite for enhanced electrocatalytic alkaline oxygen evolution reaction

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1. Chemials

All chemicals can be used directly without extra purification. Nickel nitrate hexahydrate ($Ni(NO_3)_2·6H_2O$), thioacetamide (CH_3CSNH_2, referred to as TAA), Naphthol solution (Nafion) were purchased from Shanghai Aladdin Reagent Co., Ltd. Urea (H_2NCONH_2), potassium hydroxide (KOH) and anhydrous ethanol (C_2H_6O) were purchased from China Pharmaceutical Chemical Reagents Co., Ltd. Cerium nitrate hexahydrate ($Ce(NO_3)_3·6H_2O$) was purchased from Shanghai McLean Biochemical Co., Ltd. Glucose ($C_6H_{12}O_6$) was purchased Jiangsu Qiangsheng Functional Chemistry Co., Ltd.

2. Characterization

The structure and properties of the samples were systematically analyzed by various characterization methods. The crystal structure of the material was analyzed by X-ray diffractometer (XRD) (D/max 2500 PC). The surface morphology of the samples was observed by field emission scanning electron microscopy (SEM) (Supra 55), and the elemental composition was analyzed by energy dispersive X-ray spectroscopy (EDS). High-resolution transmission electron microscopy (HR-TEM, JEM-2000) was used to obtain the microstructure information of the material. In addition, the chemical states of the elements on the catalyst surface were studied by X-ray photoelectron spectroscopy (XPS) (PHI-5000 VPIII), and the molecular vibration modes of the materials were analyzed by Raman spectroscopy (LabRAM HR Evolution).

3. Preparation of electrodes

In the process of constructing the working electrode, 2 mg catalyst powder(In this article refers to Ni_3S_4) was first suspended in500 μL water-ethanol mixed solution (v/v, 3:1), and 20 μL of 5 wt % Nafion solution was introduced as a binder, and the standard drop coating method was used to complete the preparation. Continuous ultrasonic dispersion for 2 h to form a uniform catalyst ink. Subsequently, 5 μL catalyst ink was uniformly dropped on the surface of a glassy carbon electrode with a geometric area of 0.07 cm^2 using a micropipette. Upon natural drying at room temperature, the catalyst loading was measured to be 0.275 mg cm^{-2}.

4. Electrochemical measurements

All electrochemical tests were performed using the DH7000 electrochemical testing system (manufactured by Donghua Testing Technology Co., Ltd.). The three-electrode configuration was used for performance testing in 1 M KOH electrolyte. The working electrode was a glassy carbon electrode coated with a catalyst, the counter electrode was a graphite rod, and the reference electrode was Hg/HgO. The LSV curve was infrared compensated by 90 %, and the solution resistance was 1.5Ω. Linear sweep voltammetry (LSV) was used to evaluate the catalytic performance of the electrocatalyst. The corresponding Tafel slope was extracted by Tafel equation analysis of the polarization curve. Electrochemical impedance spectroscopy (EIS) tests were performed in the frequency range of 0.01 Hz ~ 100 kHz using a sinusoidal disturbance amplitude of 5 mV. To quantify the capacitance of the electric double layer (C_{dl}) and the active electrochemical surface area (ECSA), CV measurements were systematically executed within the non-faradaic region at scan rates ranging from 20 to 120 mV s^{-1} (20 mV s^{-1} per interval), enabling calculation of interfacial charge storage characteristics through capacitive current analysis. The electrochemical stability of the electrode was evaluated through chronopotentiometry under a constant current density of 10 mA cm^{-2}, with the catalyst demonstrating sustained performance over a 20 hour durability test. Through thermodynamic relationships, all measured potentials are converted to reversible hydrogen electrode (RHE) scales by the following relationships:

$$E\ vs.\ \text{RHE} = E\ vs.\ \text{SCE} + 0.098 + 0.0591 \times \text{pH},$$

where pH corresponds to the electrolyte environment during testing.

5. Figures

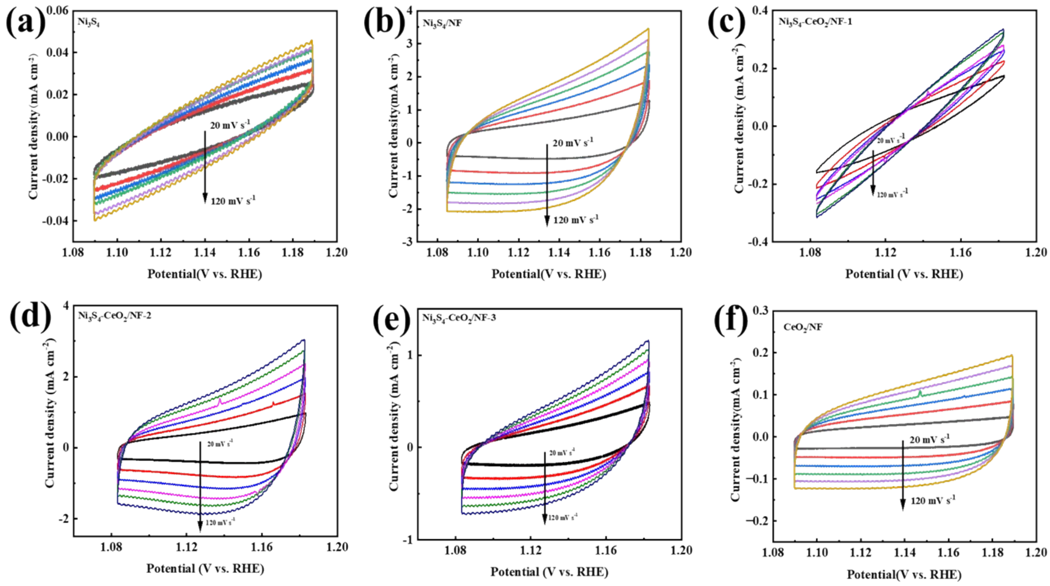
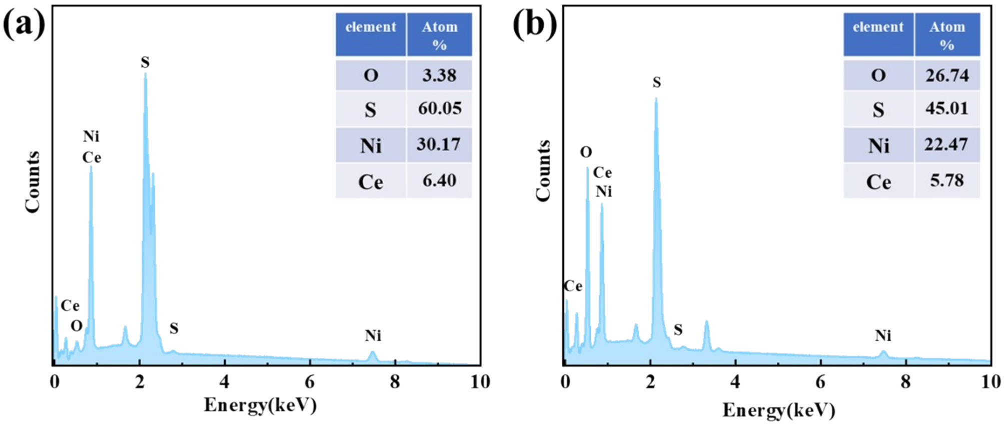


Fig. S1. (a) CV curves of Ni_3S_4, (b) Ni_3S_4/NF, (c) Ni_3S_4-CeO_2/NF-1, (d) Ni_3S_4-CeO_2/NF-2, (e) Ni_3S_4-CeO_2/NF-3, (f) CeO_2/NF.



(a)

element	Atom %
O	3.38
S	60.05
Ni	30.17
Ce	6.40

(b)

element	Atom %
O	26.74
S	45.01
Ni	22.47
Ce	5.78

Fig. S2. The EDS spectra of Ni_3S_4-CeO_2/NF-2 before(a) and after 20h reaction (b).