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Surface-Reconstructed FeOOH@CoFeOS/NF Architectures: Leveraging Nano-layered Stacking for Accelerated Oxygen Evolution

Kinetic

Materials and methods

Thiourea (CH₄N₂S, \geq 99 %) was purchased from Shenyang Chemical Reagent Factory. Fe(NO₃)₃·9H₂O was obtained from China National Medicines Corporation. Co(NO₃)₂·6H₂O, was obtained from Shanghai Aladdin Biochemical Technology Factory. NH₄F was provided by Liaoning Quanrui Reagent Factory. All reagents in this experiment are of AR grade and can be used directly without further purification. NF was supplied from Shenzhen Hanbo Environmental Protection Equipment Factory. NF (1.5×2.4 cm²) was washed successively in 3.0 M HCl, anhydrous ethanol, and deionized water by ultrasonication for 10 min, and then dried at 45 ° C for 6 h.



Figure S1. (a) XRD images of CoFeO/NF. (b) XRD images of FeSO/NF. (c) XRD images of CoSO/NF.



Figure S2. LSV curves of CoFeO, FeOS, CoOS and CoFeOS.



Figure S3. Cyclic voltammogram curves of (a) NF, (b) CoFeSO/NF, (c) FeOOH/NF and (d) FeOOH@CoFeSO/NF at the different scan rates from 10 to 100 mV s⁻¹ in the potential range of open Circuit Potential. Time V vs. RHE;



Fig. S4. Polarization curves of different catalysts at a scan rate of $1 \text{ mV} \text{ s}^{-1}$ (the FeOOH@CoFeOS/NF from the lower potential toward higher potential)



Fig. S5. The XRD pattern of FeOOH@CoFeOS/NF, (b) spectras of FeOOH/NF, CoFeOS/NF, FeOOH@CoFeOS/NF (c) FT-IR spectras of FeOOH/NF, CoFeOS/NF, FeOOH@CoFeOS/NF. (after continuous operation).



Fig. S6. XPS survey spectrum of (a) O 1s (b) Fe 2p, (c) S 2p, (d) Co 2p in the sample of FeOOH@CoFeOS/NF (after continuous operation).



Fig. S7. Morphology characterization: (a) SEM images of low-power FeOOH@CoFeOS/NF. (b) SEM images with high magnification FeOOH@CoFeOS/NF (after continuous operation).

After a long period of IT testing, it was found that there was no significant change in the XPS spectra before and after testing, and the peak positions were roughly consistent. Among them, the micro scanning SEM image revealed that during the testing process, some of the active substances in the nanosheet stacking part fell off, and the overall morphology did not change significantly. The performance did not change much, but still had excellent performance. After analyzing the object, it was found that the material changes were not significant.



Figure S8. Equivalent circuit used for fitting the EIS data.

Catalyst	Electrolyte	η10 (mV)	Reference
FeOOH@CoFeOS/NF	1.0M KOH	212	This work
CoFe-BDC-NO ₂	1.0M KOH	292	J. Mater. Sci. Technol. 189 (2024) ⁴³
Vo-Fe-Co ₃ O ₄	1.0M KOH	231	Chem. Commun. Technol. 2024 ⁴⁴
NiFe-MOF-S@CNT	1.0M KOH	237	J. Alloys Compd. 972 (2024) ⁴⁵
Ni-NCN/CoFe-LDH	1.0M KOH	280	J. Colloid Interface Sci. 650 (2023) ⁴⁶
FeCo-Ni ₃ S ₄	1.0M KOH	230	J. Chem. Eng. 940 (2023) ⁴⁷
NiCoFe-Se/CFP	1.0M KOH	221	J. Colloid Interface Sci. 642 (2023) ⁴⁸
Fe-Co ₉ S ₈ @CoO	1.0M KOH	296	J. Hydrog. Energy 47 (2022) ⁴⁹
Co8FeS8/Fe7S8@NC	1.0M KOH	255	J. Solid State Chem. 310 (2022) ⁵⁰
Ni-Co-Fe ₃ O ₄	1.0M KOH	243	J. Electroanalytical Chemistry 940 (2023) ⁵¹
Ni ₃ S ₂ /Fe ₃ O ₄ /NF	1.0M KOH	220	J. Colloid Interface Sci. 660 (2024) 440-44852

Table S1 Comparison of OER properties of FeOOH@CoFeOS/NF with reported non-noble metal based electrocatalysts.