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# Highly Active FeCo Bimetallic Oxyhydroxide for Efficient Oxygen Evolution in Water Electrolysis

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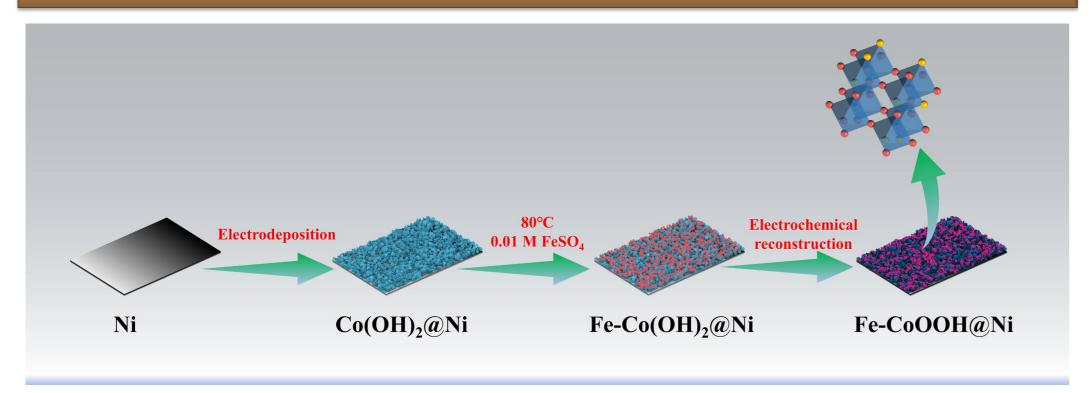
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# INTRODUCTION & AIM

The global transition toward a sustainable, carbon-neutral energy future hinges critically on the large-scale production and utilization of green hydrogen — a versatile, zero-emission energy carrier with unparalleled gravimetric energy density. Among the available pathways, water electrolysis powered by renewable electricity stands out as the most promising technology for scalable and truly green hydrogen generation. However, the widespread deployment of electrolyzers is still hampered by high system costs and efficiency losses, primarily due to the sluggish kinetics and poor durability of the oxygen evolution reaction (OER) at the anode — a complex four-electron/proton transfer process that imposes significant overpotentials and material degradation under industrially relevant operating conditions.

Inspired by these insights, we herein report a scalable, calcination-free, interfacial engineering strategy to fabricate vertically aligned Fe-doped CoOOH (Fe–CoOOH) nanosheets directly on nickel foam (Fe–CoOOH@Ni) via pulsed electrodeposition of  $Co(OH)_2$ , followed by low-temperature  $Fe^{2+}$  adsorption and controlled electrochemical activation. This approach enables precise spatial and chemical control over Fe incorporation, triggering a well-defined surface reconstruction that yields a semi-crystalline, microcrack-enriched nanostructure with optimized electronic configuration and maximized active site exposure. The resulting catalyst demonstrates exceptional OER activity ( $\eta$  = 283.7 mV @ 100 mA cm<sup>-2</sup>) and durability, driven by Fe-induced modulation of the Co valence state, reduced charge-transfer resistance, and RDS alteration. This work provides a generalizable interfacial design principle for constructing high-performance, industrially viable OER electrocatalysts through controlled surface restructuring.

#### **METHOD**



**Electrochemical Restructuring to Fe-CoOOH@Ni:** 

The Fe-CoOOH@Ni electrode was synthesized through a three-step process: first, nickel foam was cleaned and used as a substrate; second, a Co(OH)<sub>2</sub> precursor was electrodeposited onto it via cyclic voltammetry in a Co(NO<sub>3</sub>)<sub>2</sub> solution; third, iron was incorporated by immersing the electrode in an FeSO<sub>4</sub> solution at 80 °C, followed by electrochemical restructuring in 1M KOH via CV to form the final Fe-CoOOH@Ni catalyst.

#### **RESULTS & DISCUSSION**

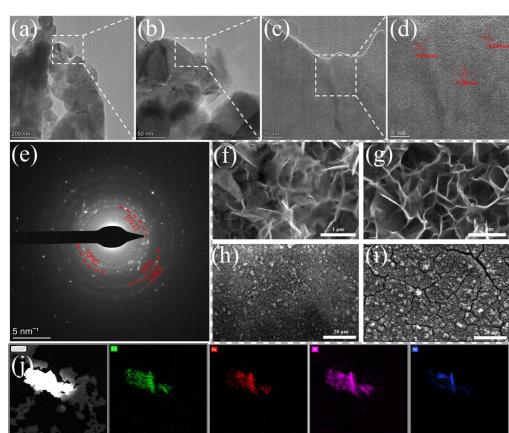


Figure1.Morphological and structural characterization of restructured Fe-CoOOH@Ni.(a-d)HRTEM (e)SAED (f-i) SEM (j) TEM-EDS

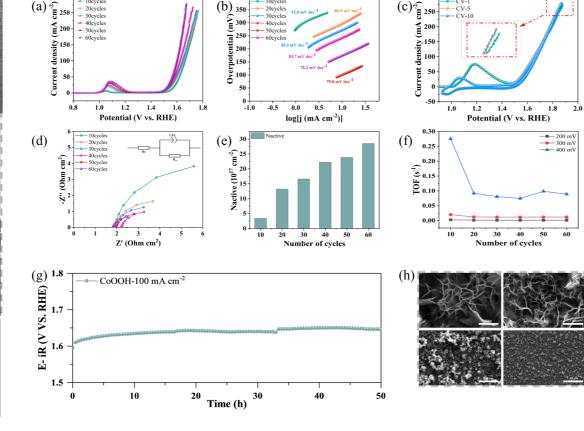


Figure 2. Optimization of CoOOH@Ni electrodeposition cycles. (a) LSV (b) Tafel (c) CV (d) EIS (e) Active site density as a function of cycle number. (f) TOF (g) Chronopotentiometric stability at 100 mA cm<sup>-2</sup>. (h) Post-stability SEM image.

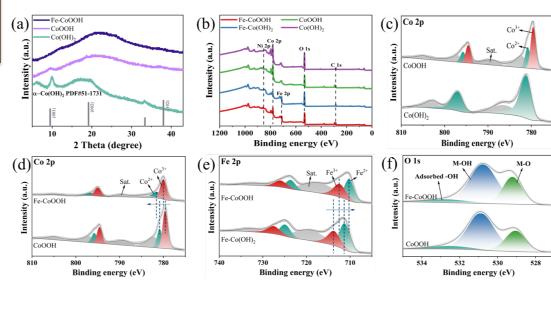
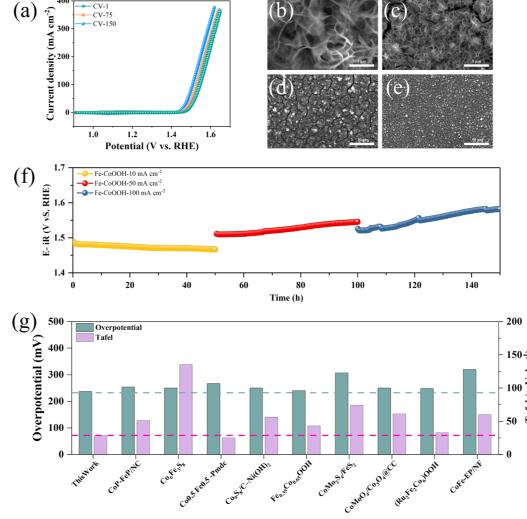


Figure 3. Phase evolution and chemical state analysis.

(a) XRD patterns of Co(OH)<sub>2</sub>, CoOOH, and Fe-CoOOH.

(b) XPS survey spectra. High-resolution XPS spectra of (c) Co 2p in Co(OH)<sub>2</sub> and CoOOH, (d) Co 2p in CoOOH and Fe-CoOOH, (e) Fe 2p in Fe-Co(OH)<sub>2</sub> and Fe-CoOOH, and (f) O 1s in CoOOH and Fe-CoOOH.



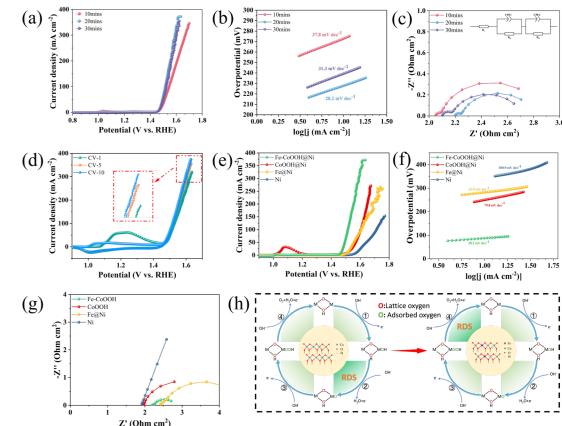


Figure 4. Effect of Fe immersion time on OER performance. (a) LSV (b) Tafel (c) EIS (d) CV. (e,f) Comparison of LSV and Tafel performance among CoOOH, Fe@Ni, bare Ni, and Fe-CoOOH@Ni. (g) EIS comparison of all catalysts. (h) Schematic diagram of RDS change

Figure 5. Long-term stability and benchmarking. (a) LSV curves before and after 150 CV cycles. (b—e) Post-stability SEM images at different magnifications. (f) Chronopotentiometric stability test at 10, 50, and 100 mA cm<sup>-2</sup> for 150 hours. (g) Overpotential at 10 mA cm<sup>-2</sup> versus Tafel slope comparison with recently reported Co-based OER catalysts.

## CONCLUSION

In summary, we have developed a facile, scalable, and calcination-free interfacial engineering strategy to construct highly efficient and durable Fe-doped CoOOH nanosheets (Fe-CoOOH@Ni) directly on nickel foam via in situ electrochemical restructuring. Mechanistic investigations reveal that the superior performance stems from the synergistic effects of Fe doping and in situ restructuring: (i) Fe incorporation modulates the local electronic structure of Co sites, promoting the formation of catalytically critical high-valent Co⁴⁺ species; (ii) the restructuring process generates abundant surface defects and microcracks, enhancing mass transport and exposing more active sites; and (iii) the optimized Co-O-Fe interfacial configuration alters the OER rate-determining step and facilitates rapid charge transfer kinetics. This work not only provides a high-performance, costeffective electrocatalyst for green hydrogen production but also establishes a generalizable paradigm for designing advanced electrocatalytic interfaces through controlled in situ surface restructuring. The insights into the dynamic evolution of surface chemistry and electronic structure during activation offer valuable guidance for the rational development of next-generation energy conversion materials.

### FUTURE WORK / REFERENCES

[1] H. Chu, R. Li, P. Feng, D. Wang, C. Li, Y. Yu, M. Yang, Ligands Defect-Induced Structural Self-Reconstruction of Fe–Ni–Co-Hydroxyl Oxides with Crystalline/Amorphous Heterophase from a 2D Metal–Organic Framework for an Efficient Oxygen Evolution Reaction, ACS Catalysis, 14 (2024) 1553-1566.

[2] B. Deng, J. Shen, J. Lu, C. Huang, Z. Chen, F. Peng, Y. Liu, Ru doping triggering reconstruction of cobalt phosphide for coupling glycerol electrooxidation with seawater electrolysis, Journal of Energy Chemistry, 100 (2025) 317-326.