Electronic Supplementary Information

Experimental Section

Materials: Potassium chromate (K₂CrO₄) was purchased from Sinopharm Chemical Reagent Co.,Ltd. The FeCl₂·6H₂O was purchased from Beijing Chemical Corp (China). Nafion (5 wt%) solution, RuCl₃·3H₂O was purchased from Sigma-Aldrich Chemical Reagent Co., Ltd. Hydrochloric acid (HCl), ethanol was purchased from Aladdin Ltd. (Shanghai, China). All reagents were used as received. The water used throughout all experiments was purified through a Millipore system. NF was purchased from Suzhou Taili New Energy Co., Ltd. The NF was cleaned according to the method described in a previous study.¹ Briefly, the NF (3 cm \times 2 cm \times 0.1 cm) was washed ultrasonically in acetone (15 mL) for 10 min and then aqueous HCl (3 M, 15 mL) for 10 min, followed by rinsing with water and ethanol and then air drying at 60 °C for 24 h.

Preparation of Fe-doped NiCr₂O₄ nanoparticle film on Ni foam (Fe-NiCr₂O₄/NF): Fe-NiCr₂O₄/NF was synthesized using an in situ hydrothermal method. Typically, K₂CrO₄ and FeCl₂·6H₂O were dissolved in 40 mL deionized water under mechanical stirring. Then, the resulting solution was transferred to a 50-mL Teflon-lined autoclave containing a piece of clean NF (2 cm × 3 cm). The autoclave was then heated at 180 °C for 24 h. After cooling to room temperature naturally, the Fe-NiCr₂O₄/NF precursor product was washed with distilled water and ethanol several times, and then dried in air at 60 °C for 24 h. Finally, the Fe-NiCr₂O₄/NF precursor products were annealed at 300 °C in air for 2 h for Fe-NiCr₂O₄/NF. As a contrast, Fe_{0.05}-NiCr₂O₄/NF and Fe_{0.15}-NiCr₂O₄/NF were prepared by varying the molar amount of FeCl₂ in solutions, under otherwise identical experimental conditions used for preparing Fe-NiCr₂O₄/NF. The actual Fe:Ni ratios in the products were determined by ICP-MS. The synthesis method of NiCr₂O₄/NF is the same as that of Fe-NiCr₂O₄/NF, except that there is no FeCl₂ in solutions.

*Preparation of RuO*₂ *electrode:* RuO₂ catalyst was prepared according to reported method.¹ In brief, 0.01 mol of RuCl₃·3H₂O was dissolved in 100 mL water and stirred

for 10 min at 100 °C. Then 1 mL KOH solution (1.0 M) was added to the solution and stirring for 45 min at 100 °C. After that, the solution was centrifuged for 10 minutes and filtered. The precipitate was washed several times with water and then dried for 5 h at 80 °C. The dry product was calcined in air at 300 °C for 3 h to obtain RuO₂. To prepare RuO₂ loaded electrodes, 20 mg RuO₂ and 10 μ L 5 wt% Nafion solution were dispersed in 1 mL 1:1 v water/ethanol solvent by 30 min sonication to form an ink finally.

Characterizations

Powder XRD data were acquired on a RigakuD/MAX 2550 diffractometer with Cu K α radiation (λ =1.5418 Å). SEM measurements were carried out on a XL30 ESEM FEG scanning electron microscope at an accelerating voltage of 20 kV. TEM measurements were performed on a HITACHI H-8100 electron microscopy (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV. XPS data were acquired on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the excitation source. *Electrochemical measurements:* Electrochemical measurements were performed with a CHI 660D electrochemical analyzer (CH Instruments, Inc., Shanghai) in a standard three-electrode system using Fe-NiCr₂O₄/NF (0.5 cm \times 0.5 cm \times 0.1 cm) as the working electrode, a graphite plate as the counter electrode and an Hg/HgO electrode as the reference electrode. Electrochemical characterization of the Fe-NiCr₂O₄/NF catalysts was carried out in 1.0 M KOH electrolytes. Optical photographs of the equipment shows in Fig. S. All potentials measured were calibrated to RHE using the following equation:

$$E (RHE) = E (Hg/HgO) + 0.098 V + 0.059 \times pH$$
 (E-1)

All electrolytes were saturated by oxygen bubbles before and during the experiments. The long-term durability test was performed using chronopotentiometric measurements. Electrochemical data were corrected for the uncompensated series resistance R_s , which was determined through fitting of AC impedance data to a modified Randles circuit. The value of R_s was 2.7 in 1.0 M KOH. The potential was determined by the following equation:

$$E_{\text{corrected}} = E_{\text{uncorrected}} - i \times R_{\text{s}}$$
(E-2)

Where i is the current.

Tafel plots calculation: The Tafel plots are employed to evaluate the OER catalytic kinetics and fitted with the following equation:

$$\eta = b \log j + a \tag{E-3}$$

Where *j* is the current density and b is the Tafel slope.

TOF calculation: The TOF is quantified the concentration of active site and calculated by the following equation:

$$TOF = \frac{jA}{4Fm}$$
(E-4)

Where *j* is current density (A cm⁻²) at defined overpotential; A is the geometric area of the testing electrode; 4 indicates the mole of electrons consumed for evolving one mole O_2 from water; F is the Faradic constant (96485 C mol⁻¹); m is the number of active sites (mol), which can be extracted from the linear relationship between the oxidation peak currents and scan rates by the following equation:

slope =
$$\frac{n^2 F^2 m}{4RT}$$
 (E-5)

Where n is the numbers of electron transferred; R and T are the ideal gas constant and the absolute temperature, respectively.

Determination of FE: The oxygen generated at anode was confirmed by gas chromatography (GC) analysis and measured quantitatively by using a calibratedpressure sensor to monitor the pressure change in the anode compartment of a H-type electrolytic cell. The FE was calculated by comparing the amount of experimentally measured oxygen generated by potentiostatic anodic electrolysis with theoretically calculated oxygen (assuming 100% FE). GC analysis was carried out on GC-2014C (Shimadzu Co.) with thermal conductivity detector and nitrogen carrier gas. Pressure data during electrolysis were recorded using a CEM DT-8890 Differential Air Pressure Gauge Manometer Data Logger Meter Tester with a sampling interval of one point per second.



Fig. S1. XRD patterns of the sample after K_2CrO_4 and $FeCl_2$ mixing (raw material), Fe-NiCr₂O₄/NF precursor.



Fig. S2. EDX spectrum for Fe-NiCr₂O₄.



Fig. S3. SEM image of bare NF.



Fig. S4. SEM image of Fe-NiCr₂O₄/NF.



Fig. S5. XPS survey spectrum for Fe-NiCr₂O₄.





Optical

photograph of OER catalyzed by Fe-NiCr₂O₄/NF.



Fig. S7. (a) XRD patterns and (b) SEM image of RuO_2 .



Fig. S8. SEM images of (a) $Fe_{0.05}$ -NiCr₂O₄/NF, (b) Fe-NiCr₂O₄/NF, and (c) $Fe_{0.15}$ -NiCr₂O₄/NF. (d) Corresponding LSV curves. HRTEM images of (e) $Fe_{0.05}$ -NiCr₂O₄, (f) Fe-NiCr₂O₄, and (g) $Fe_{0.15}$ -NiCr₂O₄. (h) Corresponding XRD patterns.



Fig. S9. LSV curves for Fe-NiCr₂O₄/NF in 30% KOH, 1.0 M KOH and 1.0 PBS.



Fig. S10. TEM image of Fe-Ni Cr_2O_4 after water oxidation.



Fig. S11. (a) CVs for Fe-NiCr₂O₄/NF under different scan rates from 10 to 50 mV s⁻¹ in 1.0 M KOH. (b) Linear relationship of the peak currents *vs.* scan rates. (c) TOF values for Fe-NiCr₂O₄/NF at different fixed overpotentials.



Fig. S12. The amount of oxygen theoretically calculated and experimentally measured versus time for Fe-NiCr₂O₄/NF in 1.0 M KOH.

Table S1. Comparison of OER performance for Fe-Ni Cr_2O_4 /NF with other Nibased OER catalysts in alkaline media.

Catalyst	Electrolyte	Tafel slope	j (mA	η at the	Ref.
		$(mV dec^{-1})$	cm ⁻²)	corresponding j	
				(mV)	
Ni-NiO	1.0 M KOH	90	10	370	2
Ni-NiO-CNT	1.0 M KOH	80	10	320	2
NiO _x	1.0 M NaOH	-	10	420	3
NiFeO _x	1.0 M NaOH	-	10	350	3
NiCo@NiCoO _x	1.0 M KOH	31	10	295	4
Fe _{0.5} Ni _{0.5} Co ₂ O ₄	1.0 M KOH	27	10	350	5
Ni ²⁺ /MnO ₂	1.0 M KOH	60	10	400	6
MnNi _x O _x	0.1 M KOH	-	10	430	7
Ni ₃ FeN-NPs	1.0 M KOH	46	10	280	8
Fe-CoP/Ti	1.0 M KOH	67	10	230	9
NiFe/NF	1 M KOH	28	80	270	10
	0.1 M KOH	33	20	270	10
Fe-NiSe/FeNi		65	50	245	11
foam		03	100	264	11
^s Au/NiFe LDH	1.0 M KOH	-	10	237	12
Fe-NiCr ₂ O ₄ /NF	1.0 M KOH	57	20	228	This
			500	318	work

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