

Supporting Information

Porous Nickel-Iron Selenide Nanosheets as Highly Efficient

Electrocatalysts for Oxygen Evolution Reaction

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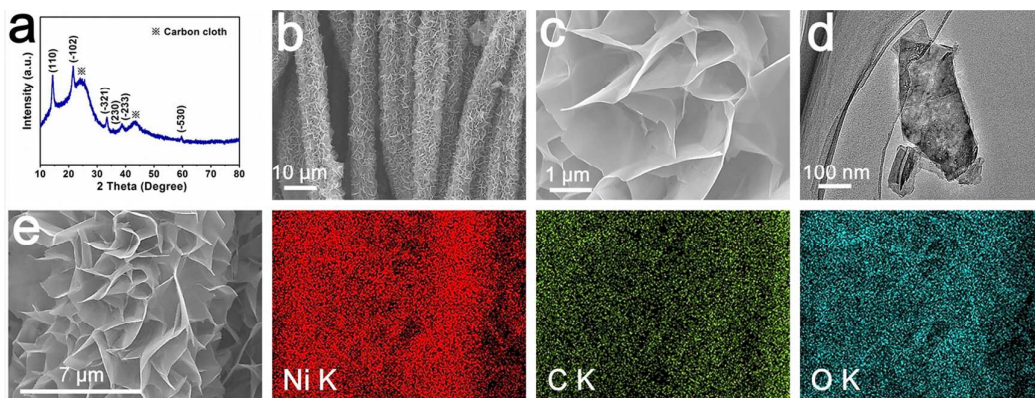


Figure S1. XRD pattern (a), SEM images (b, c), TEM image (d), SEM image and elemental mapping images (e) of the ultrathin Ni-based nanosheet precursor.

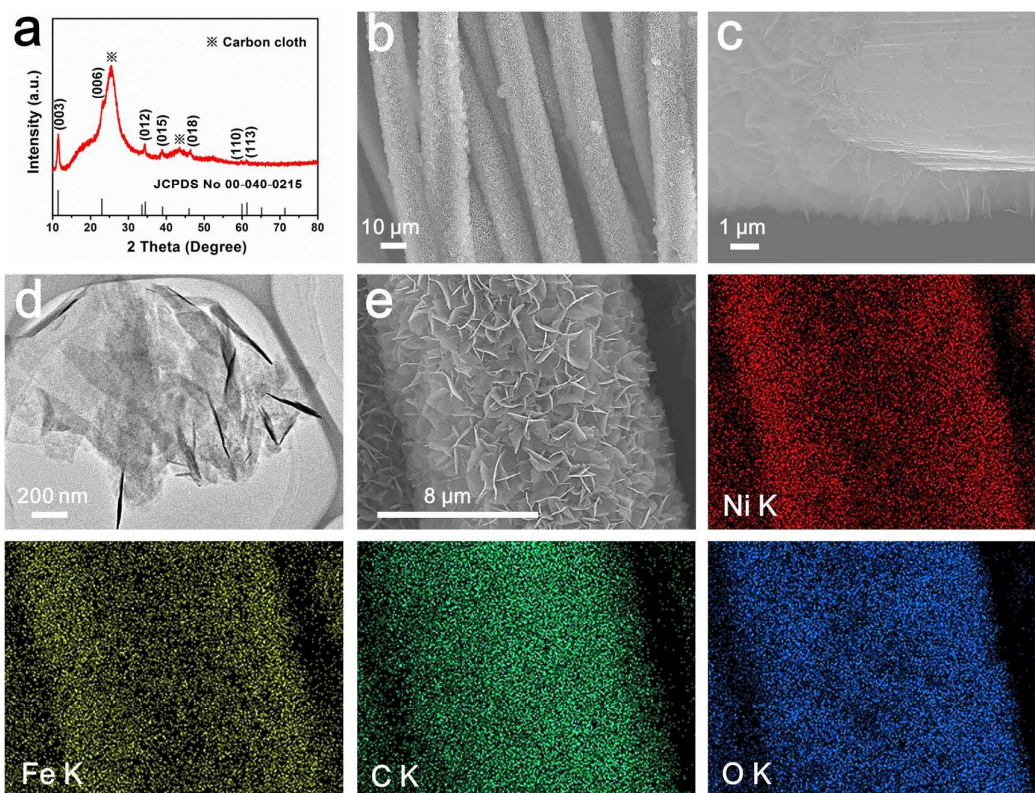


Figure S2. XRD pattern (a), SEM images (b, c), TEM image (d), SEM image and elemental mapping images (e) of the ultrathin NiFe-based nanosheet precursor.

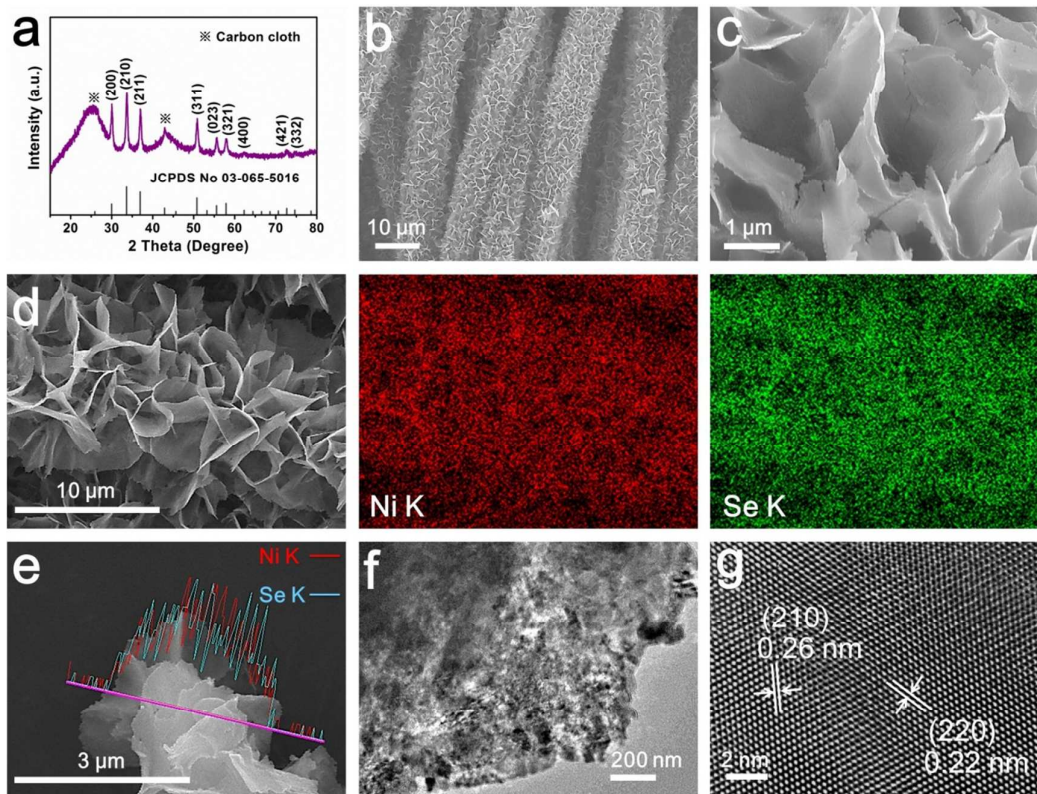


Figure S3. XRD pattern (a), SEM images (b, c), elemental mapping images (d), EDS line scans (e), TEM image (f), and HRTEM image (g) of the porous NiSe₂ nanosheets.

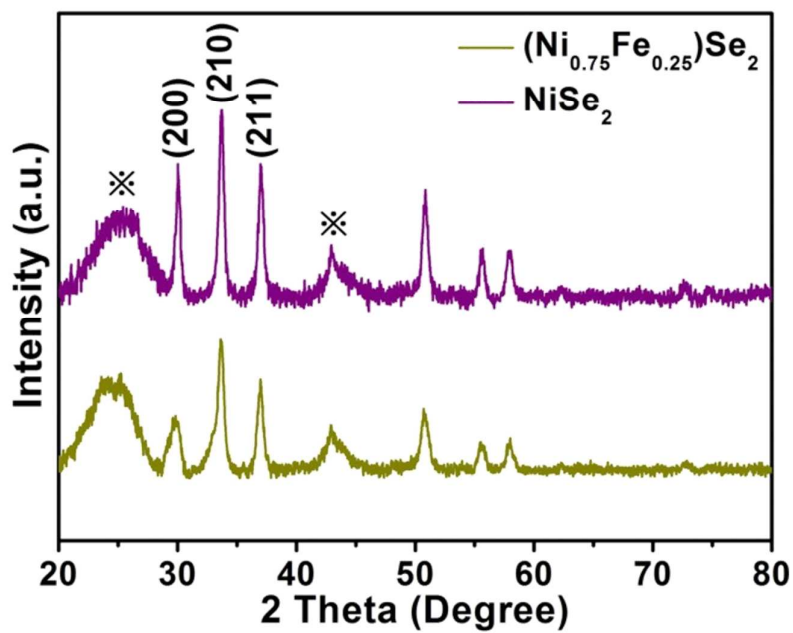


Figure S4. XRD patterns of the porous (Ni_{0.75}Fe_{0.25})Se₂ nanosheets, and the porous NiSe₂ nanosheets.

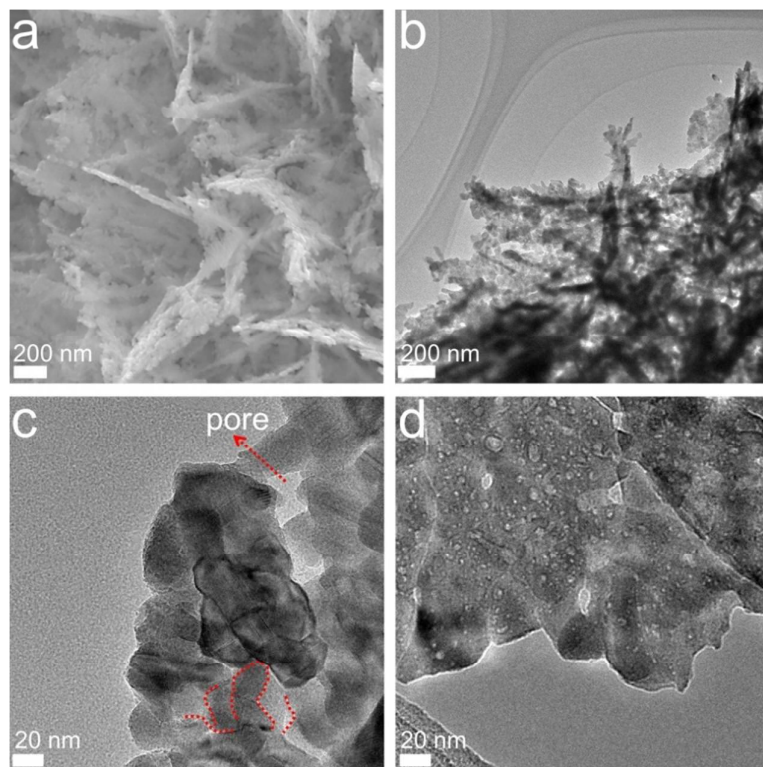


Figure S5. High-resolution SEM image (a), TEM image (b), and (c) HRTEM image of the porous $(\text{Ni}_{0.75}\text{Fe}_{0.25})\text{Se}_2$ nanosheet. TEM image (d) of the porous NiSe_2 nanosheet.

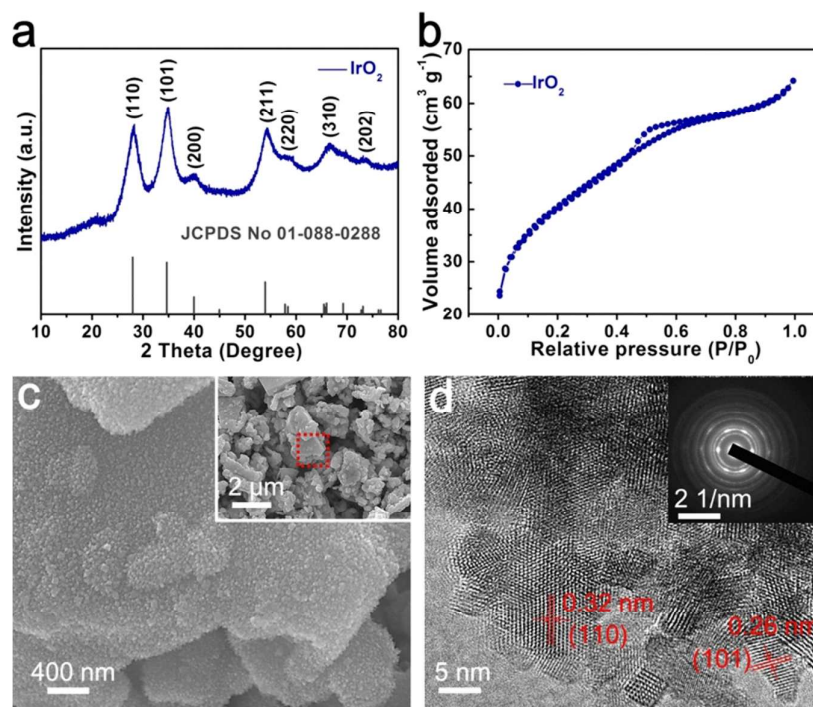


Figure S6. XRD pattern (a), Nitrogen adsorption-desorption isotherm (b), SEM images (c), HRTEM image and SAED pattern (d) of IrO_2 catalyst.

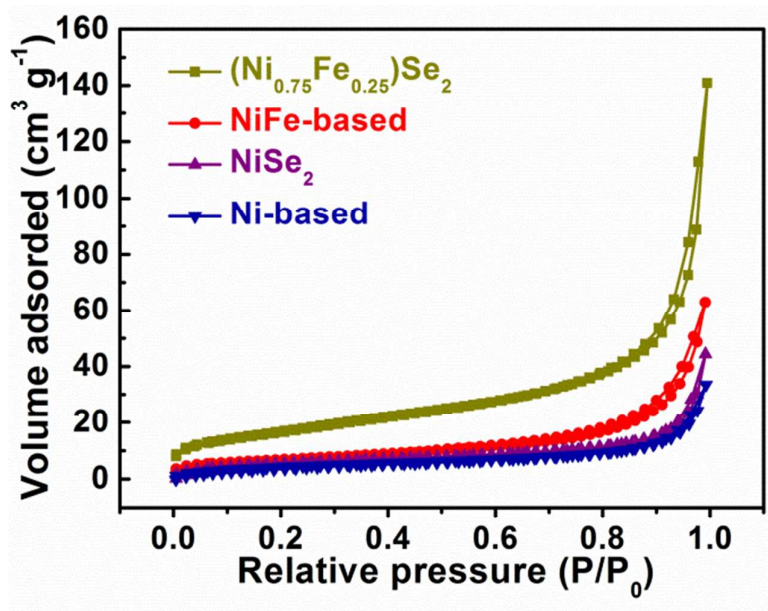


Figure S7. Nitrogen adsorption-desorption isotherm of porous $(\text{Ni}_{0.75}\text{Fe}_{0.25})\text{Se}_2$ nanosheets, NiFe-based nanosheet precursor, porous NiSe_2 nanosheets, and Ni-based nanosheet precursor.

Table S1. ICP results of NiFe-based nanosheet precursor, porous $(\text{NiFe})\text{Se}$ nanosheets, and porous NiSe_2 nanosheets.

Sample	Ni (wt.%)	Fe (wt.%)	Se (wt.%)	Element molar ratios
NiFe-based	5.204	1.712	NA	2.89:1
$(\text{NiFe})\text{Se}$	3.749	1.224	14.782	2.91:1:8.54
NiSe_2	1.091	NA	3.236	1:2.21

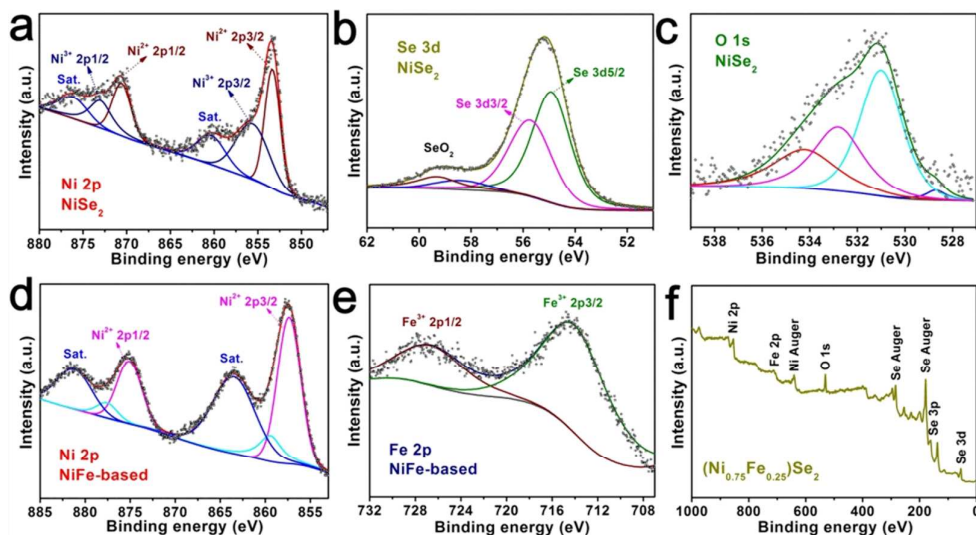


Figure S8. High-resolution (a) Ni 2p, (b) Se 3d, and (c) O 1s XPS spectra for the porous NiSe₂ nanosheets. High-resolution (d) Ni 2p and (e) Fe 2p XPS spectra for the NiFe-based precursor. XPS survey spectrum for the (f) porous (Ni_{0.75}Fe_{0.25})Se₂ nanosheets.

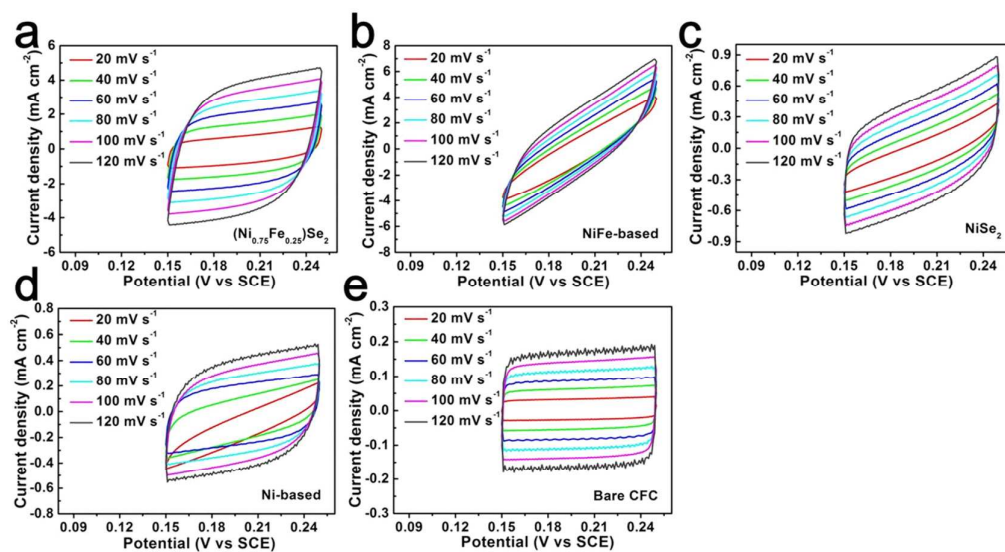


Figure S9. The double-layer capacitance measurements of the obtained (Ni_{0.75}Fe_{0.25})Se₂ (a), NiFe-based precursor (b), NiSe₂ (c), Ni-based precursor (d) and carbon fiber cloth (e) samples.

The ECSA was determined by measuring the capacitive current associated with double-layer charging from the cyclic voltammetry (CV) curves at different scan rates. A linear slope is obtained from the plot of the differences in the charging current density ($J_a - J_c$) at 0.2 V (vs. saturated calomel electrode) against the scan rate. The linear slope is twice of the double-layer capacitance (C_{dl}). The CV of the samples were tested using a three electrode system in a 0.15-0.25 V potential window with the scan rates from 20 mV s⁻¹ to 120 mV s⁻¹.

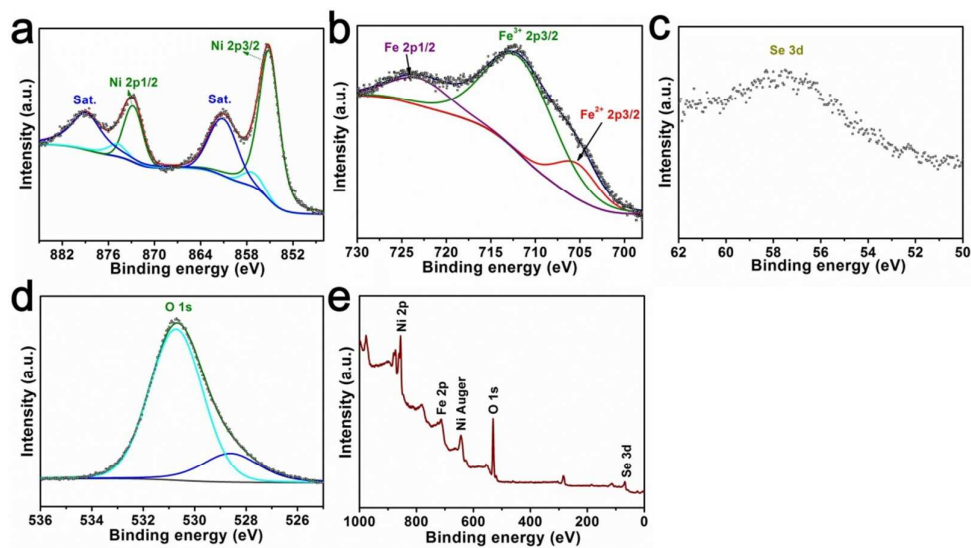


Figure S10. XPS spectra in the (a) Ni 2p, (b) Fe 2p, (c) Se 3d, and (d) O 1s regions for the $(\text{Ni}_{0.75}\text{Fe}_{0.25})\text{Se}_2$ catalyst after OER electrolysis in 1 M KOH. XPS survey spectrum of the (e) post-OER $(\text{Ni}_{0.75}\text{Fe}_{0.25})\text{Se}_2$ catalyst.

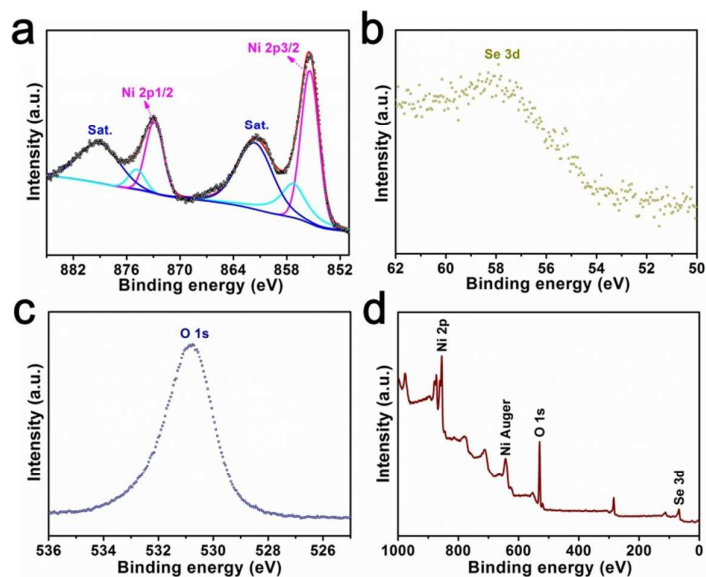


Figure S11. XPS spectra in the (a) Ni 2p, (b) Se 3d, and (c) O 1s regions for the NiSe_2 catalyst after OER electrolysis in 1 M KOH. XPS survey spectrum of the (d) post-OER NiSe_2 catalyst.

Table S2. ICP results for the porous $(\text{NiFe})\text{Se}$ nanosheets and porous NiSe_2 nanosheets after OER electrolysis.

Sample	Ni (wt.%)	Fe (wt.%)	Se (wt.%)	Element molar ratios
$(\text{NiFe})\text{Se}$	0.792	0.256	0.572	2.94:1:1.58
NiSe_2	1.465	NA	0.937	1:0.48

Table S3. The composition analysis from deconvolution of XPS spectra before and after OER test.

Catalysts (Ni _{0.75} Fe _{0.25})Se ₂	Element analysis (At.%)		
	Ni	Fe	Ni : Fe
Before OER	23.57	8.12	2.90 : 1
After OER	20.25	7.08	2.86 : 1

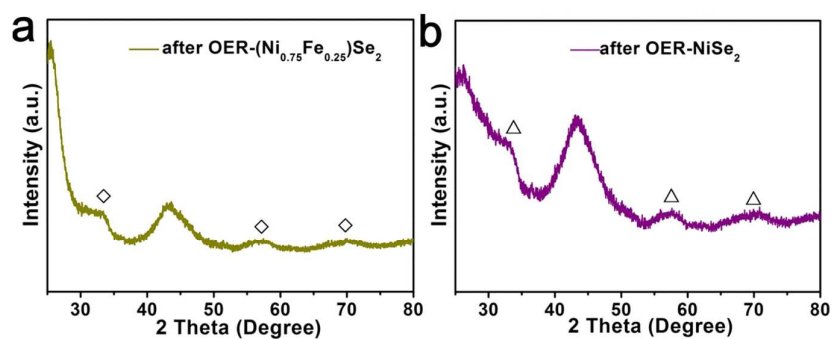


Figure S12. XRD patterns of the (Ni_{0.75}Fe_{0.25})Se₂ (a) and NiSe₂ (b) after OER electrolysis.

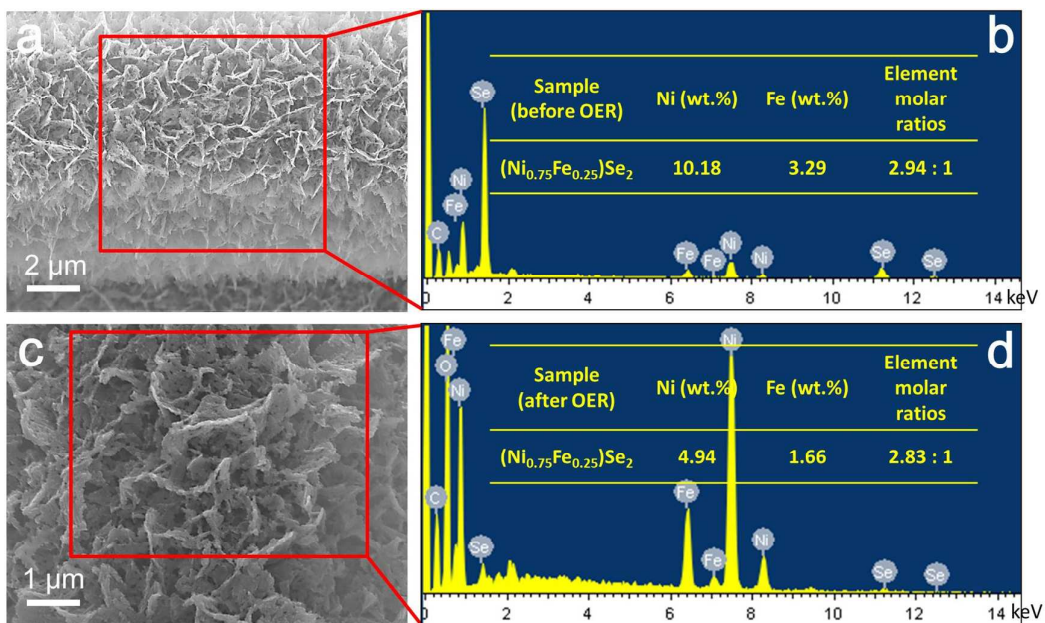


Figure S13. SEM image (a) and EDS spectrum (b) for the (Ni_{0.75}Fe_{0.25})Se₂ catalyst before OER electrolysis, SEM image (c) and EDS spectrum (d) for the (Ni_{0.75}Fe_{0.25})Se₂ catalyst after OER electrolysis.

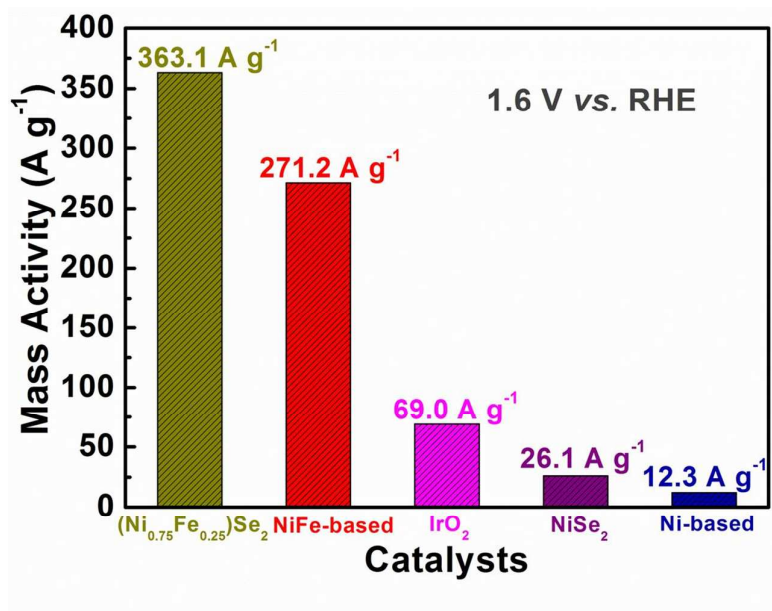


Figure S14. Mass activity based on the weight of the (Ni_{0.75}Fe_{0.25})Se₂, NiFe-based nanosheet precursor, NiSe₂, Ni-based nanosheet precursor, and IrO₂ catalysts at 1.6 V vs. reversible hydrogen electrode (RHE).

Table S4. The R_s and R_{ct} values of the samples from EIS spectra (Figure 5) simulated using the corresponding equivalent circuit.

Sample	R _s (Ω)	R ₁ (Ω)	R _{ct} (Ω)
(Ni _{0.75} Fe _{0.25})Se ₂	3.67	0.21	0.78
NiFe-based	3.65	0.19	1.22
NiSe ₂	5.05	0.39	3.39
Ni-based	4.69	1.03	21.75

Table S5. Comparison of OER activity for the (Ni_{0.75}Fe_{0.25})Se₂/CFC with some reported non-noble metal catalysts in alkaline electrolytes.

Catalysts	Mass loading mg cm ⁻²	Current density (mA cm ⁻²)	Overpotential (mV)	Tafel sloper (mV dec ⁻¹)	Double layer capacitance (mF cm ⁻²)	Electrolyte	Substrate	Reference
(Ni _{0.75} Fe _{0.25})Se ₂ nanosheets	1.5	35	255	47.2	30.2	1 M KOH	Carbon cloth	This work
NiCo ₂ S ₄ nanowires	0.4	20	280	89	31.5	1 M KOH	Carbon cloth	(1)
NiSe nanowires	2.8	20	270	64	NA	1 M KOH	Ni foam	(2)
Ni ₃ N nanosheets	--	10	256	41	NA	1 M KOH	Carbon cloth	(3)
Co ₄ N nanowires	0.8	10	257	44	365.5	1 M KOH	Carbon cloth	(4)
Co ₃ O ₄ /C nanowires	0.2	10	290	70	209.7	0.1 M KOH	Cu foil	(5)
CoP nanorods/C	0.7	10	320	71	NA	1 M KOH	Glassy carbon	(6)
CoNi(OH) _x nanotubes	0.7	10	280	77	NA	1 M KOH	Cu foil	(7)
(Ni, Co) _{0.85} Se nanotubes	5.0	10	255	79	22.9	1 M KOH	Carbon cloth	(8)
Zn _x Co _{3-x} O ₄ nanowires	1.0	10	320	51	150.7	1 M KOH	Ti foil	(9)
FeOOH/Co /FeOOH nanotubes	0.5	21	250	32	306.4	1 M NaOH	Ni foam	(10)

Additional references:

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