

Supporting Information

Ni nanotube array-based electrodes by electrochemical alloying and de-alloying for efficient water splitting

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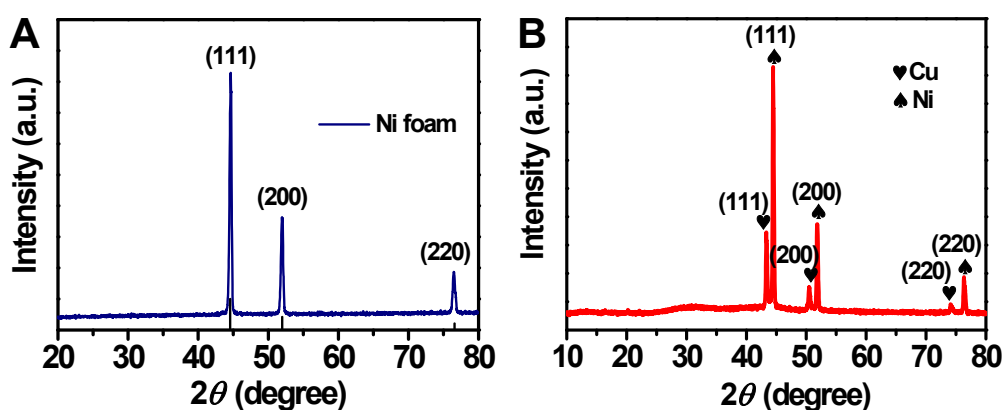


Figure S1. (A) XRD pattern of the bare Ni foam. (B) XRD pattern of NiCu alloy scraped from the FTO substrate.

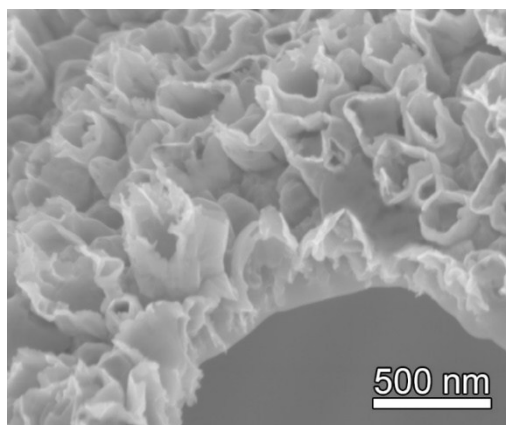


Figure S2. The cross-sectional SEM image of Ni NTAs.

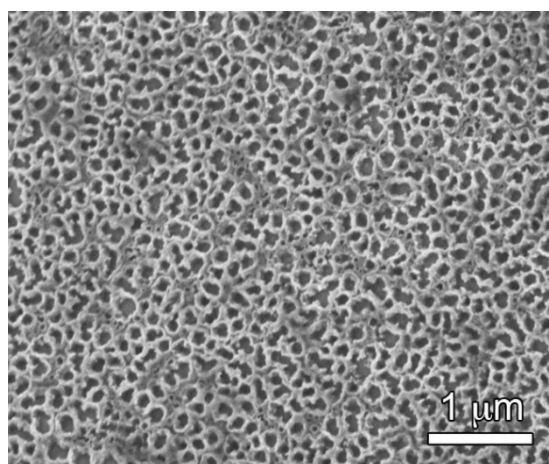


Figure S3. SEM images of Ni NTAs synthesized by 5 min cathodic electrodeposition in 0.05 M CuSO₄ solution.

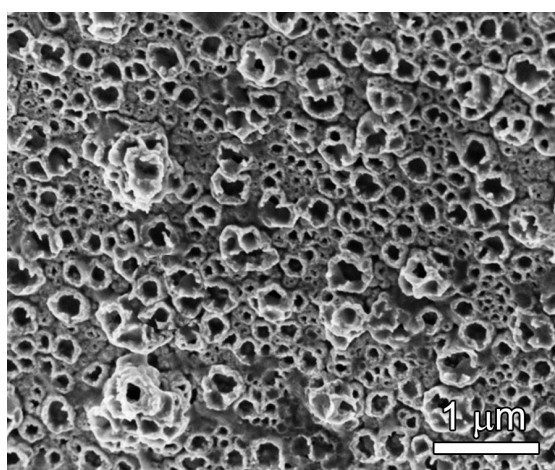


Figure S4. SEM images of Ni NTAs synthesized by 8 min cathodic electrodeposition in 0.1 M CuSO₄ solution.

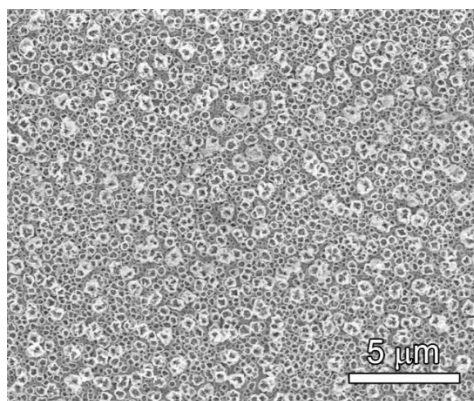


Figure S5. SEM image of Ni NTAs at an FTO substrate.

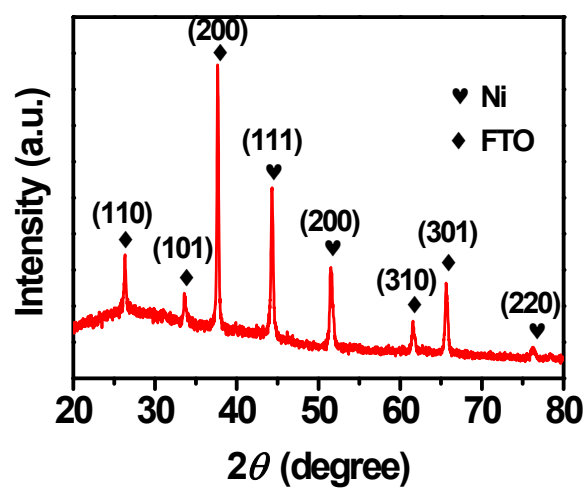


Figure S6. XRD pattern of Ni NTAs at an FTO substrate.

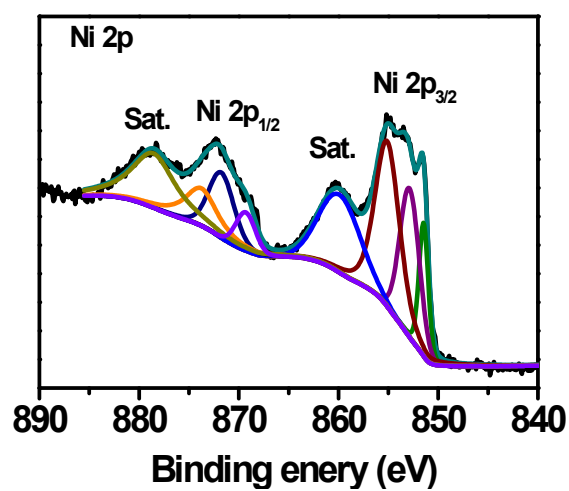


Figure S7. Ni 2p XPS of Ni NTAs at an FTO substrate.

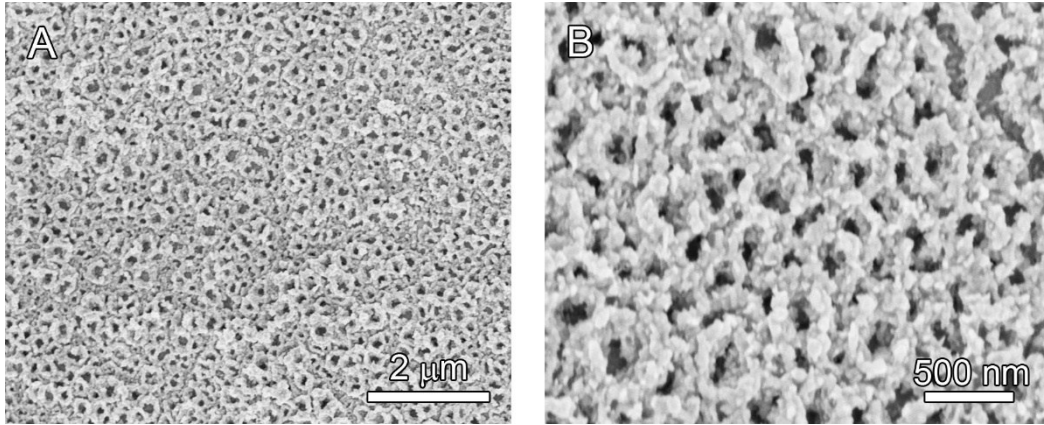


Figure S8. SEM images of NiSe₂ NTAs by cathodic deposition for 10 min.

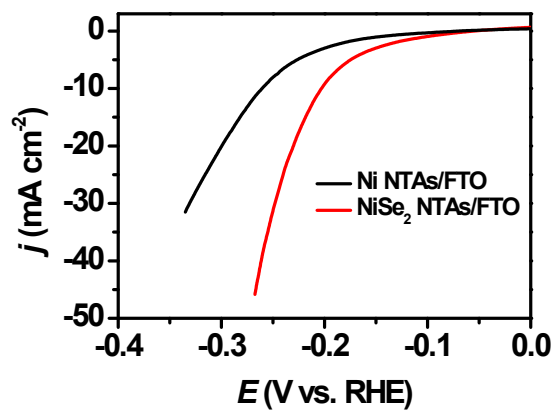


Figure S9. LSV curves for HER on Ni NTAs and NiSe₂ NTAs at an FTO substrate.

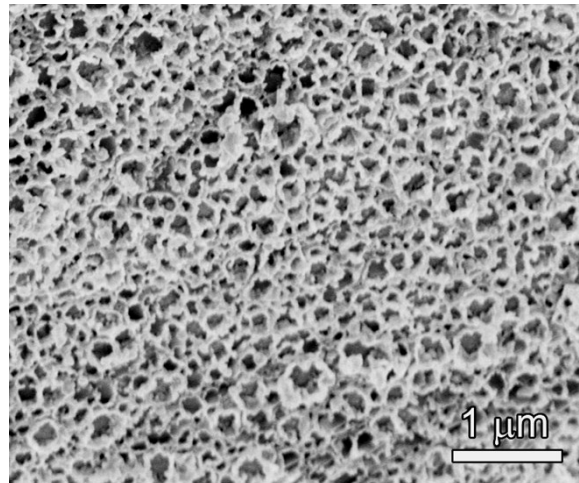


Figure S10. SEM image of NiSe₂ NTAs after electrolysis in 1 M KOH.

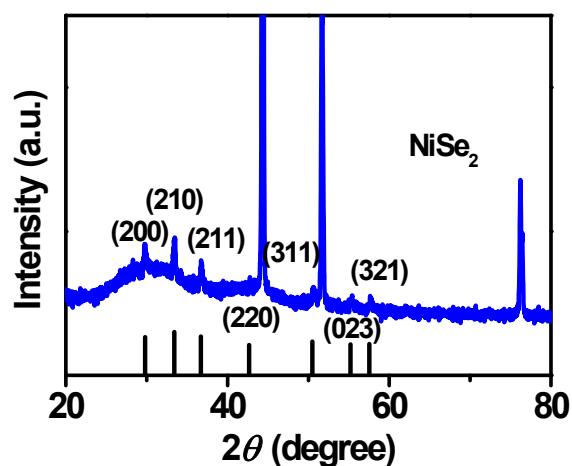


Figure S11. XRD pattern of NiSe₂ NTAs after electrolysis in 1 M KOH.

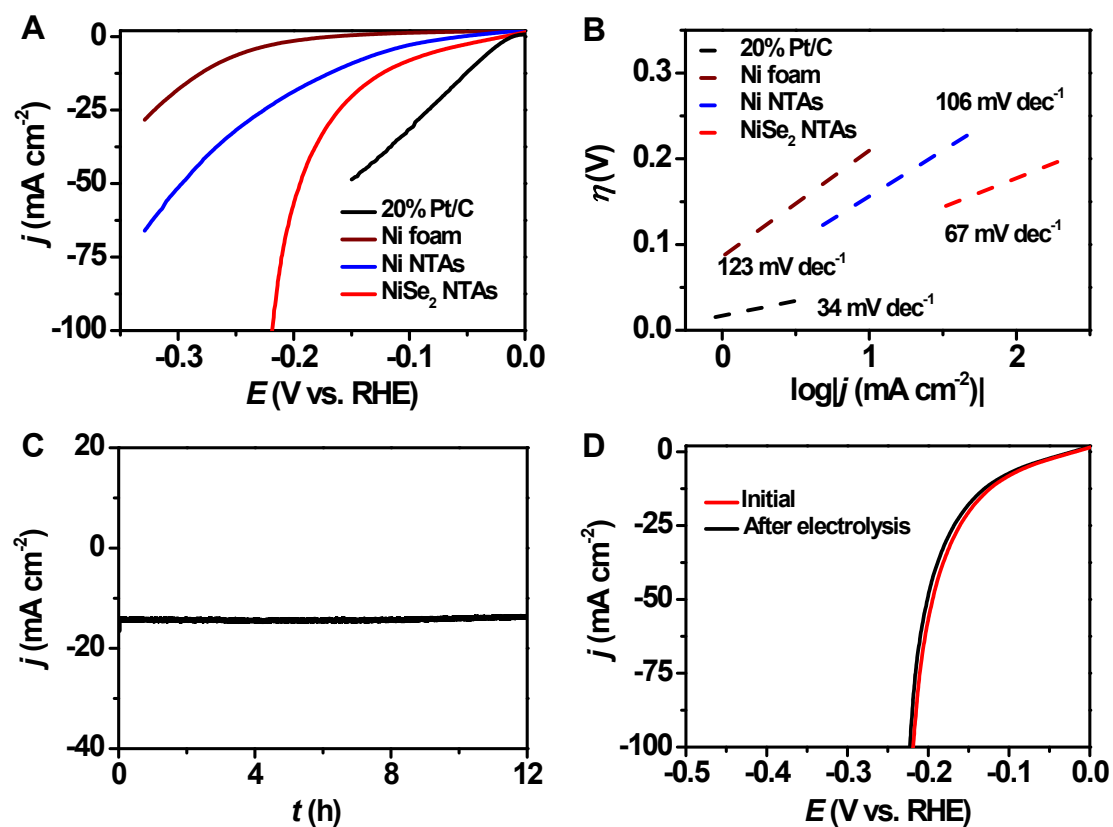


Figure S12. (A) Polarization curves and (B) Tafel plots for the HER on 20% Pt/C, Ni foam, Ni NTAs, and NiSe₂ NTAs. (C) Time-dependent current density curve of NiSe₂ NTAs under an overpotential of 160 mV. (D) HER polarization curves of NiSe₂ NTAs before and after 12 h electrolysis. All measurements were performed in 0.5 M H₂SO₄.

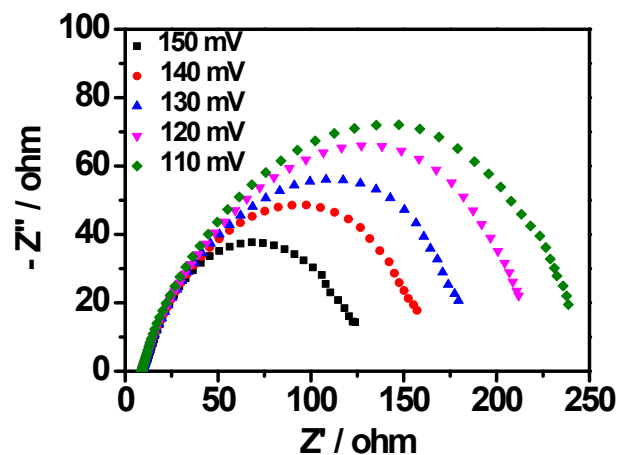


Figure S13. Nyquist plots of the NiSe₂ NTAs at various overpotentials in 1M KOH.

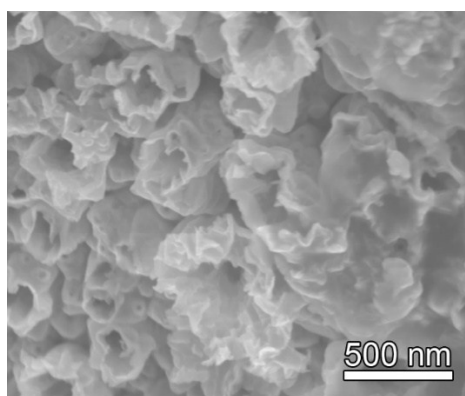


Figure S14. The cross-sectional SEM image of NiFeO_x NTAs.

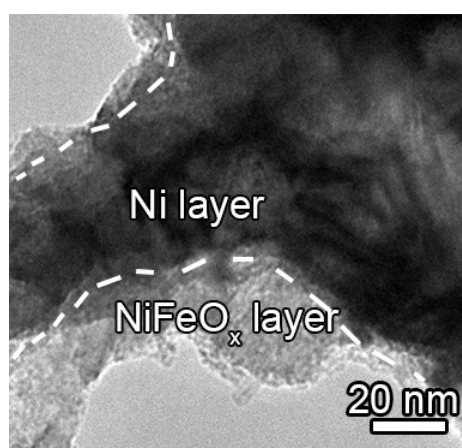


Figure S15. TEM image of scraped NiFeO_x NTAs. We note that NiFeO_x layer is uneven and its thickness ranges from a few nanometers to tens of nanometers.

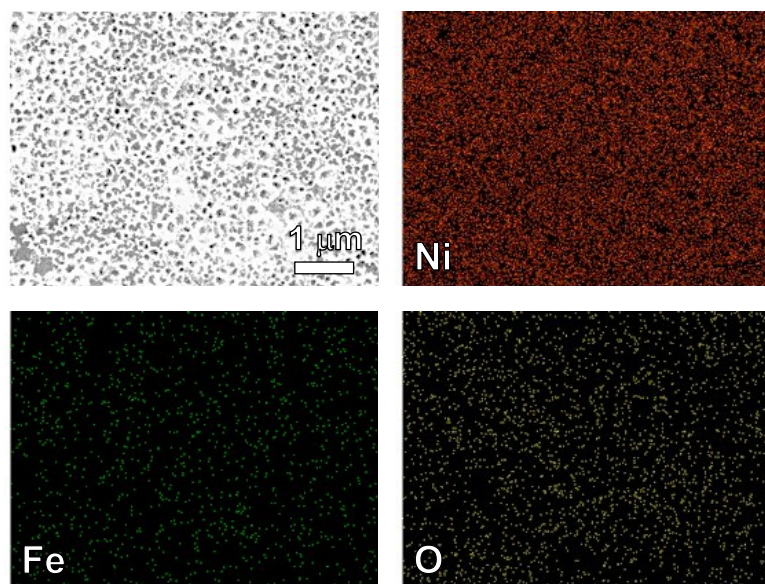


Figure S16. SEM mapping images of NiFeO_x NTAs.

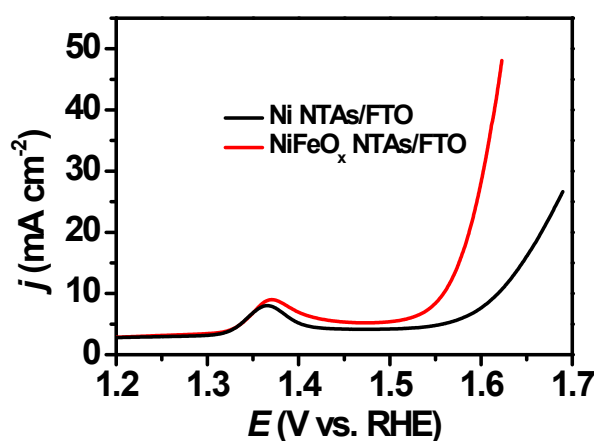


Figure S17. LSV curves of Ni NTAs and NiFeO_x NTAs at the FTO substrate.

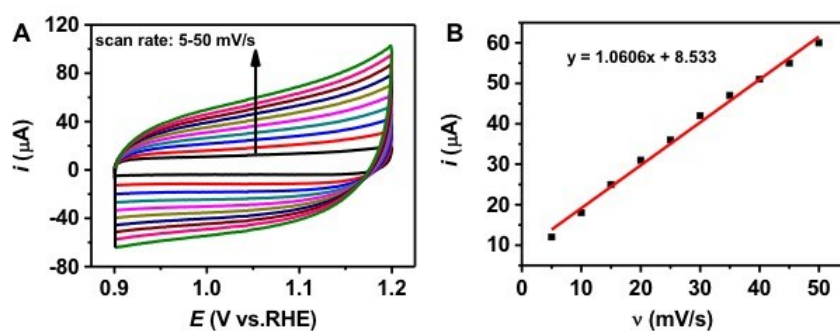


Figure S18. (A) Capacitive charging currents of the NiFeO_x NTAs electrode recorded in the non-Faradaic potential region between 0.9 V - 1.2 V at scan rates from 5 to 50 mV/s at an interval of 5 mV. (B) The anodic charging currents measured at 1.05 V

plotted as a function of scan rates.

$$\text{ECSA} = C_{\text{DL}}/0.040 = 1.0606/0.040 = 26.515 \text{ cm}^2$$

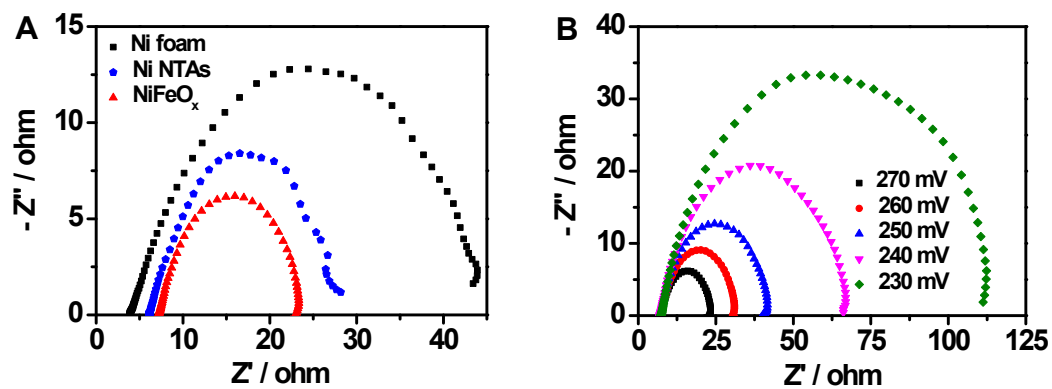


Figure S19. (A) Nyquist plots of Ni foil, Ni NTAs and NiFeO_x NTAs in 1 M KOH.

(B) Nyquist plots of NiFeO_x NTAs at various potentials in 1 M KOH.

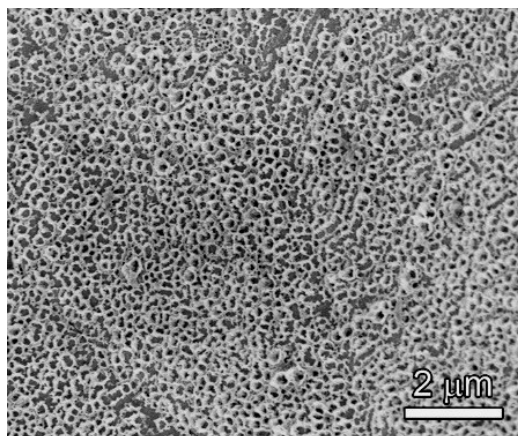


Figure S20. SEM image of NiFeO_x NTAs after electrolysis in 1 M KOH.

Table S1. Comparison of catalytic performance of various 3D NiSe₂ catalysts.

Catalysts	Electrolyte	Current density (mA cm ⁻²)	Overpotential (mV)	Tafel slope (mV dec ⁻¹)	Ref.
Porous NiSe ₂ /NF ^U	0.5 M H ₂ SO ₄	10	143	49	1
Porous NiSe ₂ /NF ^U	0.5 M H ₂ SO ₄	10	153	46	2
NiSe ₂ nanosheets/CF	0.5 M H ₂ SO ₄	10	117	32	3
NiSe ₂ nanosheets/NF	0.5 M H ₂ SO ₄	10	156	54	4
NiSe ₂ nanosheets/NF	1.0 M KOH	10	164	96	4
NiSe ₂ /CFP	0.5 M H ₂ SO ₄	10	270	68.8	5
NiSe ₂ nanowall/CC	0.5 M H ₂ SO ₄	10	145	41	6
NiSe₂ NTAs/NF	0.5 M H₂SO₄	10	98	67	This work
NiSe₂ NTAs/NF	1.0 M KOH	10	60	65	This work

NF: nickel foam; CF: carbon fiber; CFP: carbon fiber paper; CC: carbon cloth.

^U uncompensated *iR* correction.

Table S2. OER performance comparison between NiFeO_x NTAs electrode and published well-known OER systems.

Catalysts	Electrolyte (KOH)	Onset overpotential(mV)	$j @ \eta = 300$ mV (mA cm ⁻²)	Tafel slope (mV dec ⁻¹)	Ref.
Fe:Ni(OH) ₂ /NF	1.0 M	215	750	42	7
NiFeO _x /IF	1.0 M	220	1000	34	8
NiFe foam ^U	1.0 M	240	80	56	9
NiFe/NiCo ₂ O ₄ /NF	1.0 M	240	280	39	10
NiCo ₂ S ₄ /NF	1.0 M	240	20	40	11
Ni ₂ Se ₃ /NF	1.0 M	230	50	144	12
NiFeO_x	1.0 M	230	400	47	This work
NTAs/NF					

IF: iron foam; LDH: layered double hydroxide.

^U uncompensated iR correction.

Table S3. Comparison of overall water splitting performance in 1.0 M KOH for NiFeO_x NTAs||NiSe₂ NTAs with other non-noble-metal electrocatalysts.

Catalyst couples	Current density (mA cm ⁻²)	Cell voltage (V)	Ref.
Pt IrO ₂	10	1.67	13
RuO ₂ Pt/C	10	1.53	14
Ni _{2.5} Co _{0.5} Fe-LDH Ni _{2.5} Co _{0.5} Fe-LDH	10	1.62	15
Co-P/NC Co-P/NC	10	1.7	13
MoO ₂ nanosheets MoO ₂ nanosheets	10	1.53	16
CoO _x @CN CoO _x @CN	20	1.55	17
NiFeO _x NiFe-P	20	1.6	8
NiFeO_x NTAs NiSe₂ NTAs	50	1.53	This work

Supplementary References

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