

Table S1. Comparison of HER and OER activities of NiS/Ni₉S₈/NF-2 with other reported TMSs electrocatalysts

Catalyst	J (mA cm ⁻²)	Overpotential (mV)		Electrolyte	Reference
		HER	OER		
NiS/Ni ₉ S ₈ /NF-2	10	115	176	1 M KOH	This Work
Co ₃ S ₄ @MoS ₂	10	136	280	1 M KOH	[1]
Co ₃ S ₄ @FNC-Co3	10	140	250	1 M KOH	[2]
T(Ni ₃ S ₂ /MnS-O)	10	116	228	1 M KOH	[3]
FNHNs	10	140	290	1 M KOH	[4]
Fe _{0.8} Ni _{0.15} S _{1.05}	10	263	228	1 M KOH	[5]
H-Fe-CoMoS	10	137	282	1 M KOH	[6]
NiS	10	-	320	1 M KOH	[7]
Fe-Ni ₃ S ₂ /FeNi	10	-	282	1 M KOH	[8]
FeNiS ₂ NS/rGO	10	-	200	1 M KOH	[9]
CoNi-S/NS-rGO-550	10	-	290	1 M KOH	[10]
NiCoFe-PS	10	-	195	1 M KOH	[11]
Ni ₃ S ₂	10	-	256	1 M KOH	[12]
N-CoS ₂ @graphene	10	-	205	1 M KOH	[13]
Fe-MoS/carbon nanocomposites	10	321	-	1 M KOH	[14]
a-CoSx	10	182	-	1 M KOH	[15]
CoS@NiCoLDH	10	124	-	1 M KOH	[16]

Table S2. Comparison of overall water splitting activities of NiS/Ni₉S₈/NF-2 with other reported TMSs electrocatalysts

Catalysts (Cathode)	Catalysts (anode)	J (mA cm ⁻²)	Potential (V)	Reference
NiS/Ni ₉ S ₈ /NF-2	NiS/Ni ₉ S ₈ /NF-2	10	1.53	This Work
Co ₃ S ₄ @MoS ₂	Co ₃ S ₄ @MoS ₂	10	1.58	[1]
Co ₃ S ₄ @FNC-Co ₃	Co ₃ S ₄ @FNC-Co ₃	10	1.58	[2]
T(Ni ₃ S ₂ /MnS-O)	T(Ni ₃ S ₂ /MnS-O)	10	1.54	[3]
FNHNs	FNHNs	10	1.55	[4]
Fe _{0.8} Ni _{0.15} S _{1.05}	Fe _{0.8} Ni _{0.15} S _{1.05}	10	1.63	[5]
V-NiS ₂	V-NiS ₂	10	1.56	[17]
S, N-CNTs/ CoS ₂ @Co	S, NCNTs/CoS ₂ @Co	10	1.63	[18]
Ni ₃ S ₂ -FeS	Ni ₃ S ₂ -FeS	10	1.61	[19]
CoNi ₂ S ₄	CoNi ₂ S ₄	10	1.58	[20]
SW-CoS@CNT	SW-CoS@CNT	10	1.56	[21]



Figure S1. SEM image of NiC₂O₄·2H₂O/NF-2 precursor.

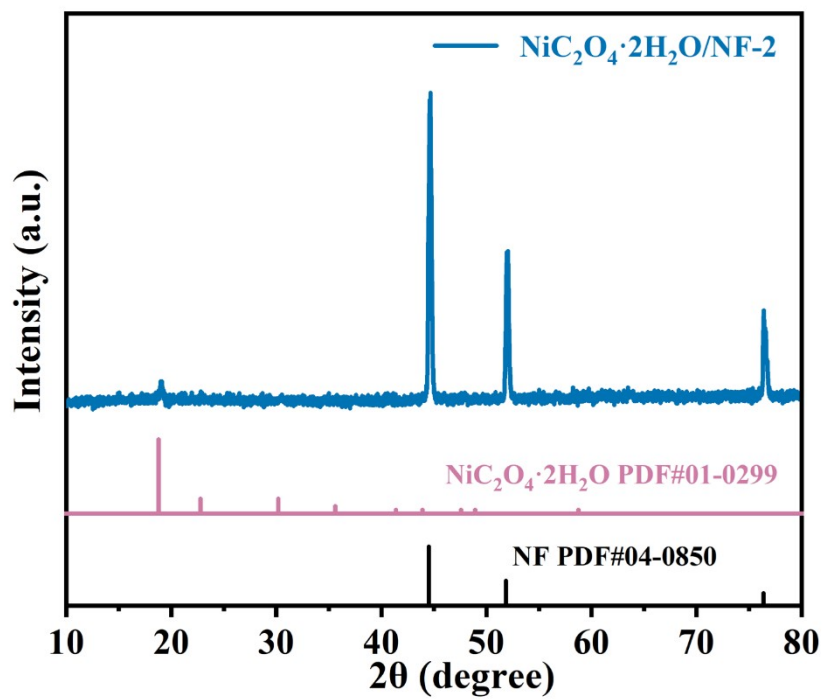


Figure S2. XRD pattern of $\text{NiC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}/\text{NF}-2$ precursor.

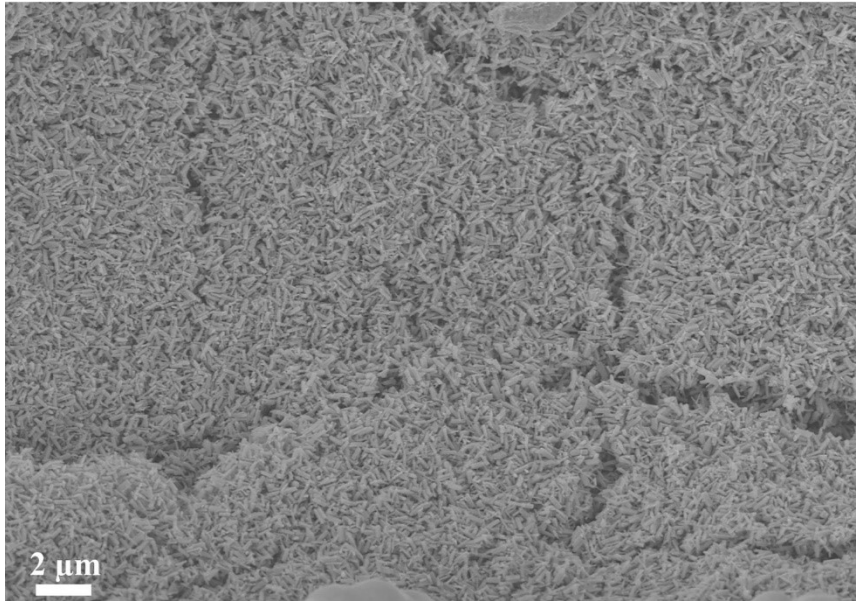


Figure S3. SEM image of NiS/Ni₉S₈/NF-2.

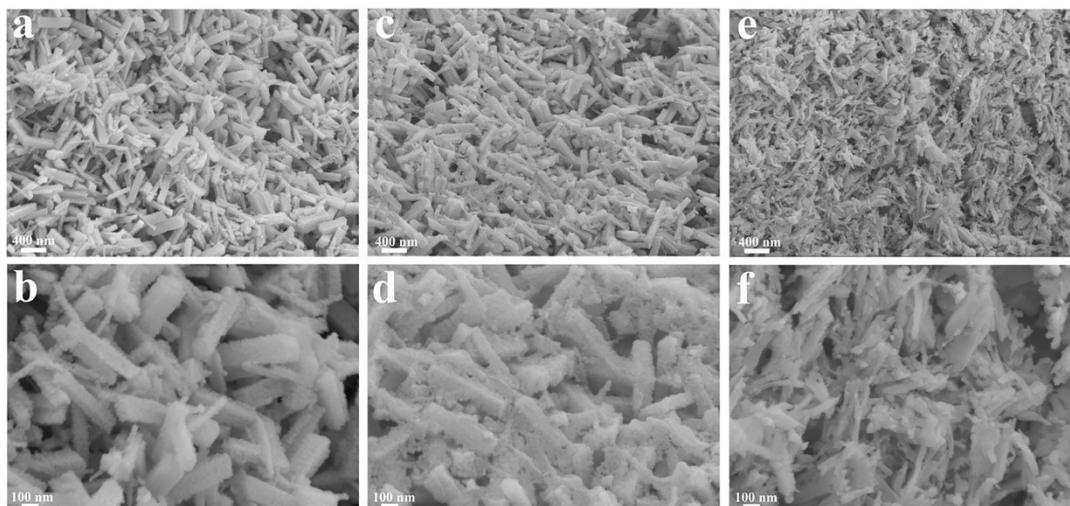


Figure S4. SEM images of (a, b) NiS/Ni₉S₈/NF-1, (c, d) NiS/Ni₉S₈/NF-3 and (e, f) NiS/Ni₉S₈/NF-4.

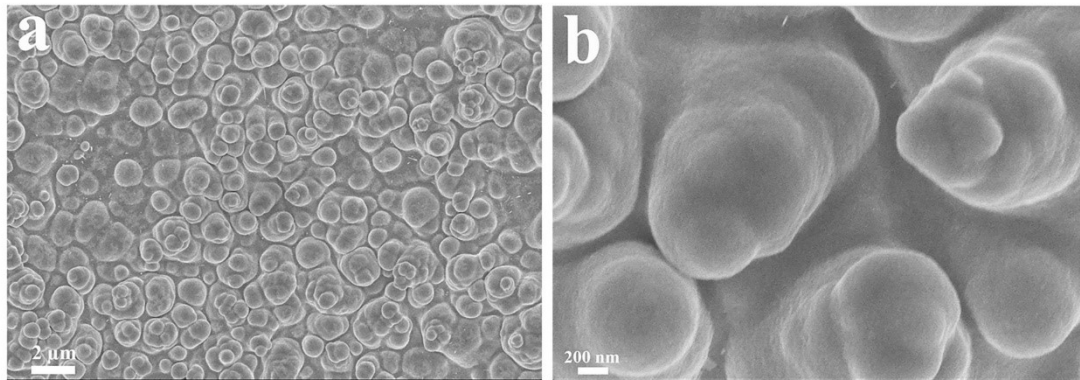


Figure S5. SEM images (a, b) of NiS/NiS₂/Ni₃S₂/NF.

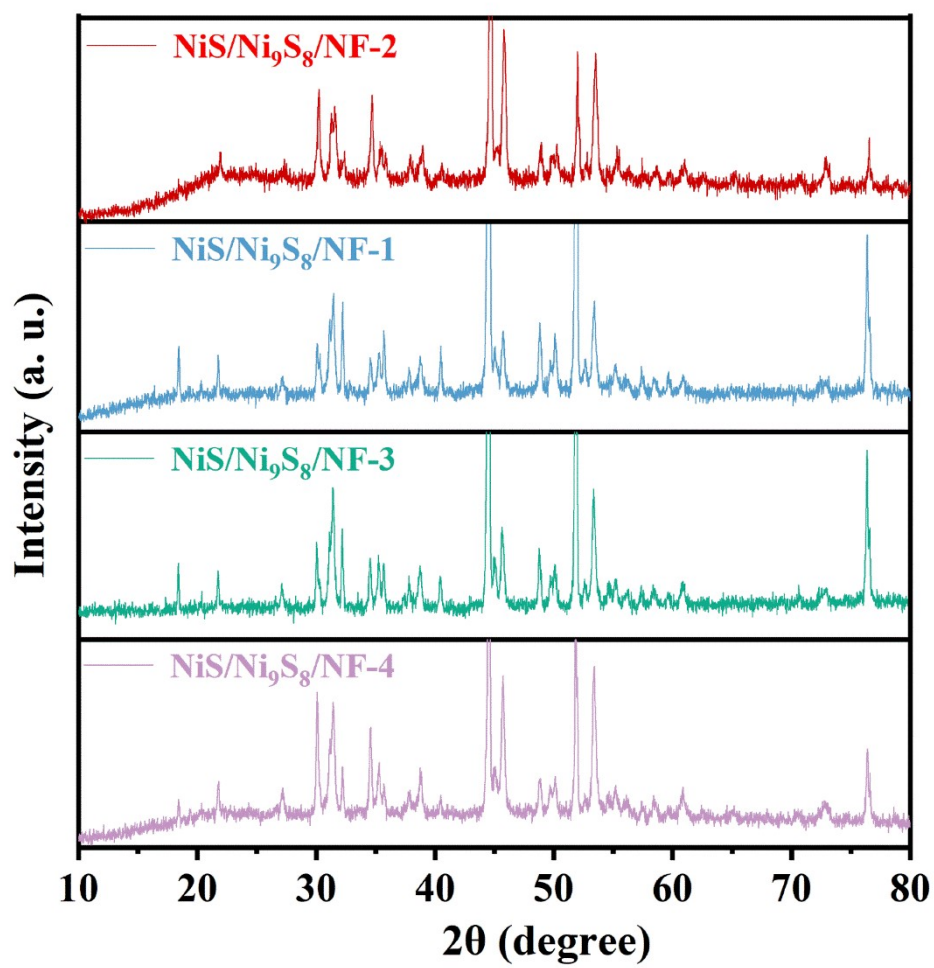


Figure S6. XRD patterns of NiS/Ni₉S₈/NF-1, NiS/Ni₉S₈/NF-3 and NiS/Ni₉S₈/NF-4.

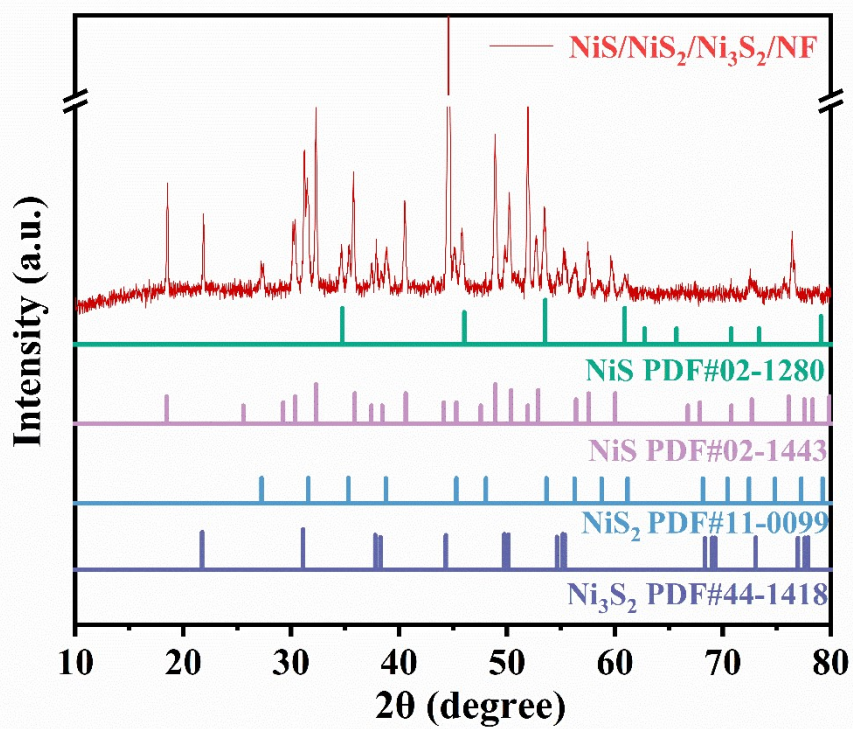


Figure S7. XRD pattern of NiS/NiS₂/Ni₃S₂/NF.

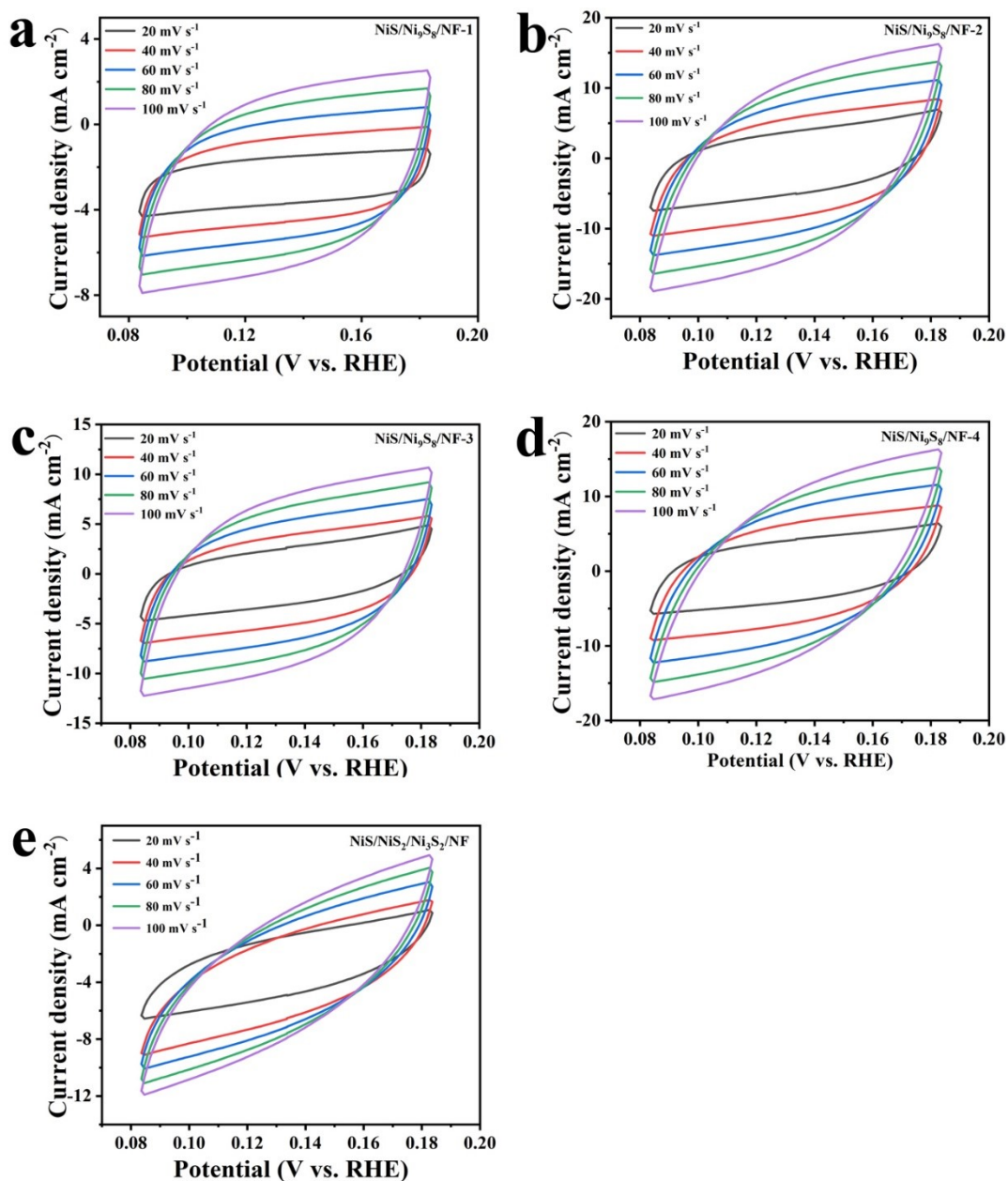


Figure S8. Cyclic voltammograms of (a) NiS/Ni₉S₈/NF-1, (b) NiS/Ni₉S₈/NF-2, (c) NiS/Ni₉S₈/NF-3, (d) NiS/Ni₉S₈/NF-4 and (e) NiS/NiS₂/Ni₃S₂/NF in -0.9 ~ -0.8 V vs. RHE at different scan rates from 20 mV s⁻¹ to 100 mV s⁻¹ in 1.0 M KOH.

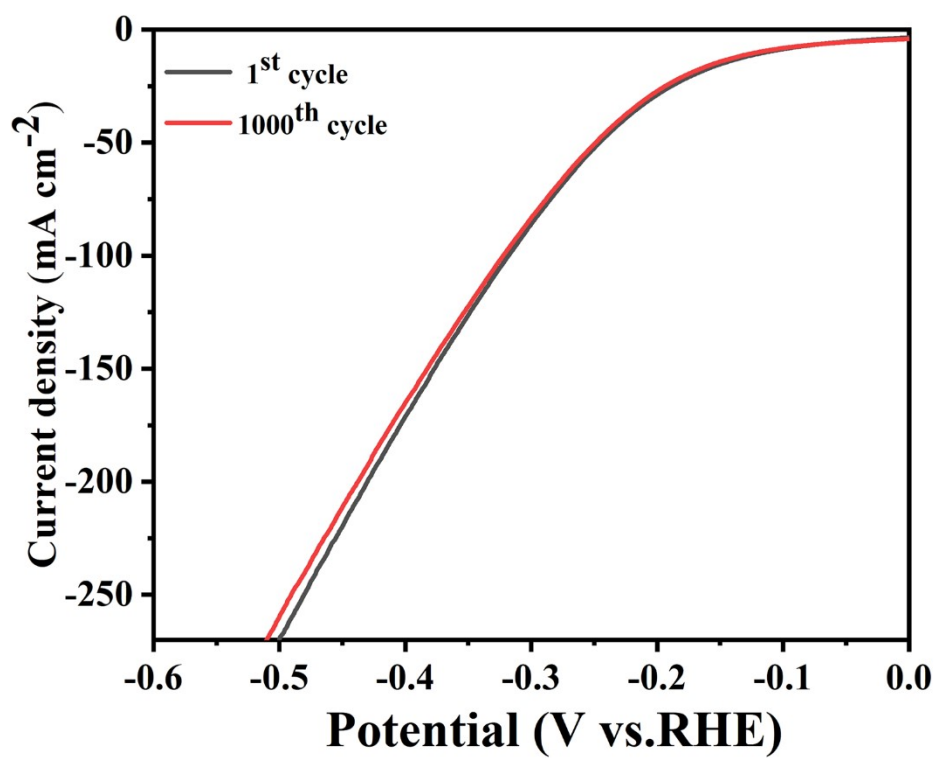


Figure S9. Polarization curves of NiS/Ni₉S₈/NF-2 before and after 1000 CV cycles.

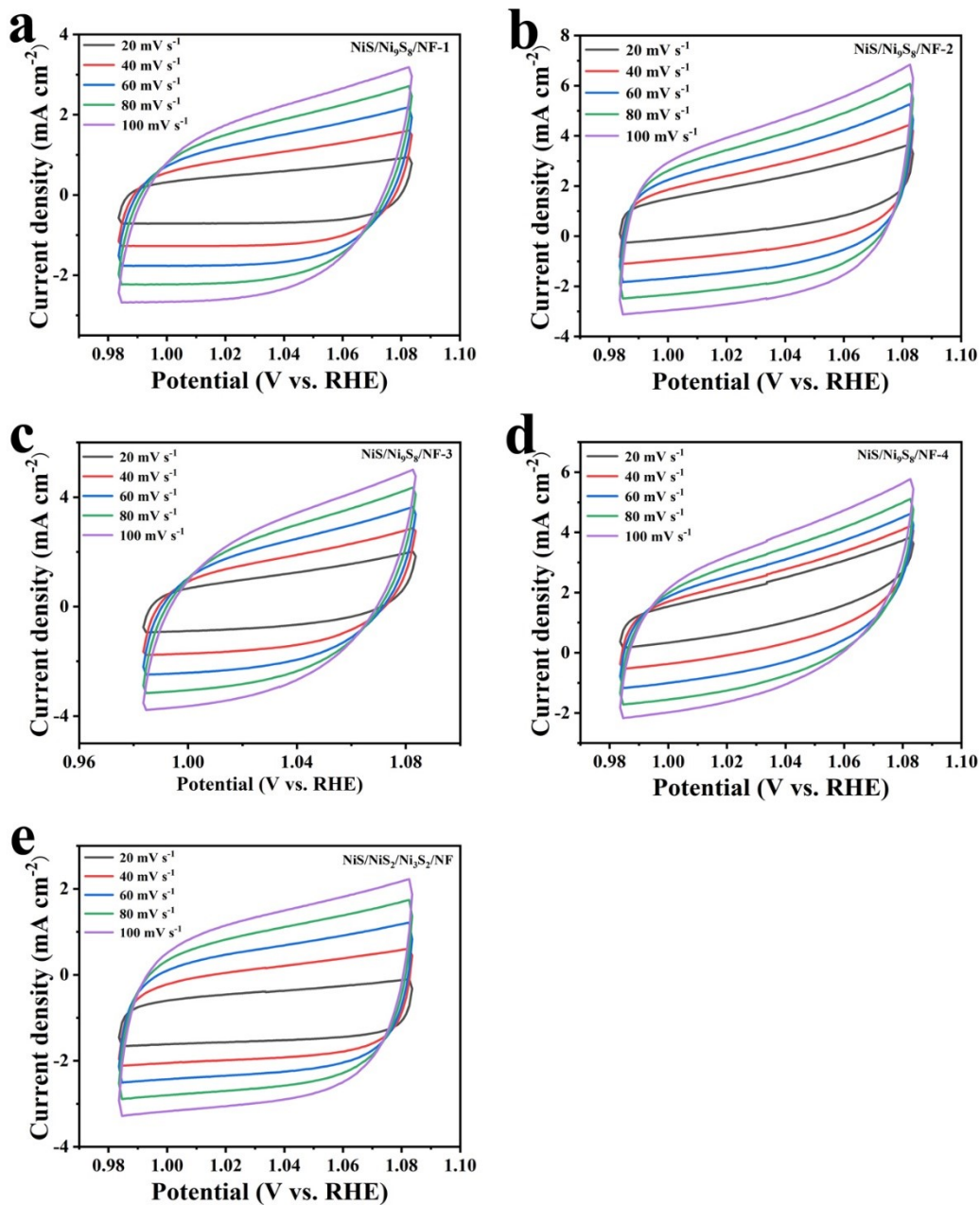


Figure S10. Cyclic voltammograms of (a) NiS/Ni₉S₈/NF-1, (b) NiS/Ni₉S₈/NF-2, (c) NiS/Ni₉S₈/NF-3, (d) NiS/Ni₉S₈/NF-4 and (e) NiS/NiS₂/Ni₃S₂/NF in -0.9 ~ -0.8 V vs.

RHE at different scan rates from 20 mV s⁻¹ to 100 mV s⁻¹ in 1.0 M KOH.

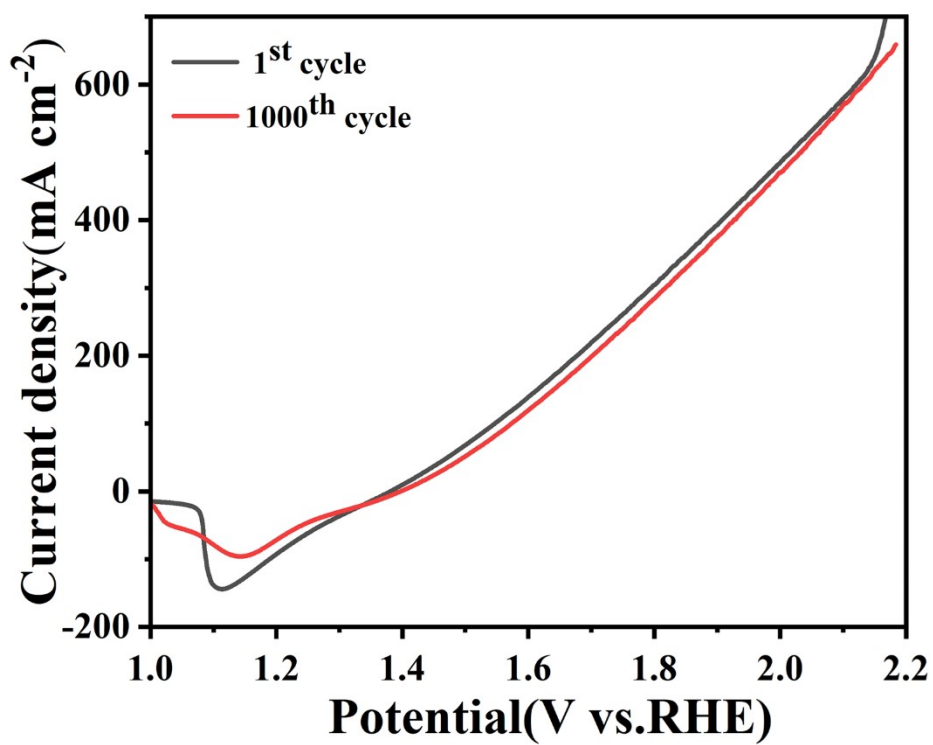


Figure S11. Polarization curves of NiS/Ni₉S₈/NF-2 before and after 1000 CV cycles.

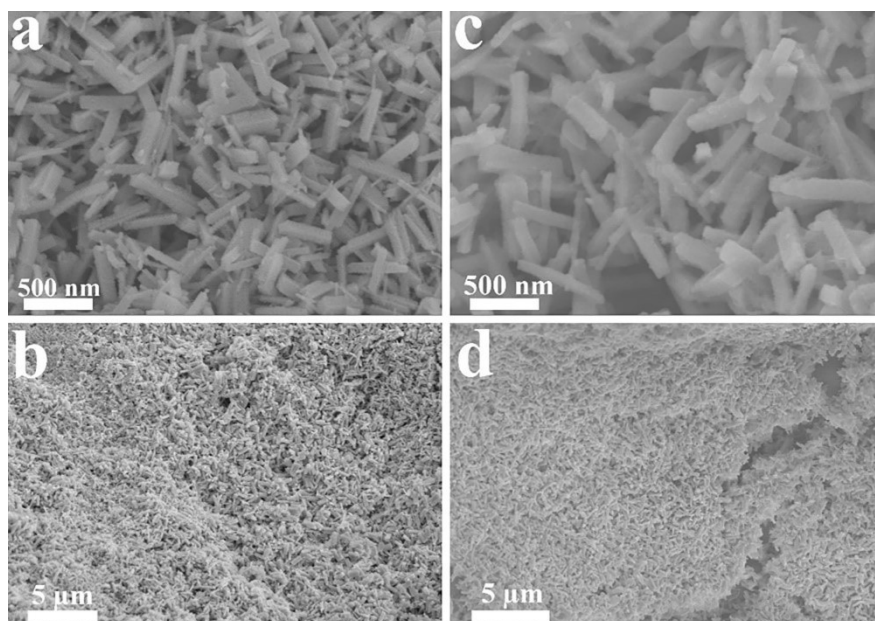


Figure S12. The SEM images of NiS/Ni₉S₈/NF-2 sample after (a,b) HER and (c,d) OER.

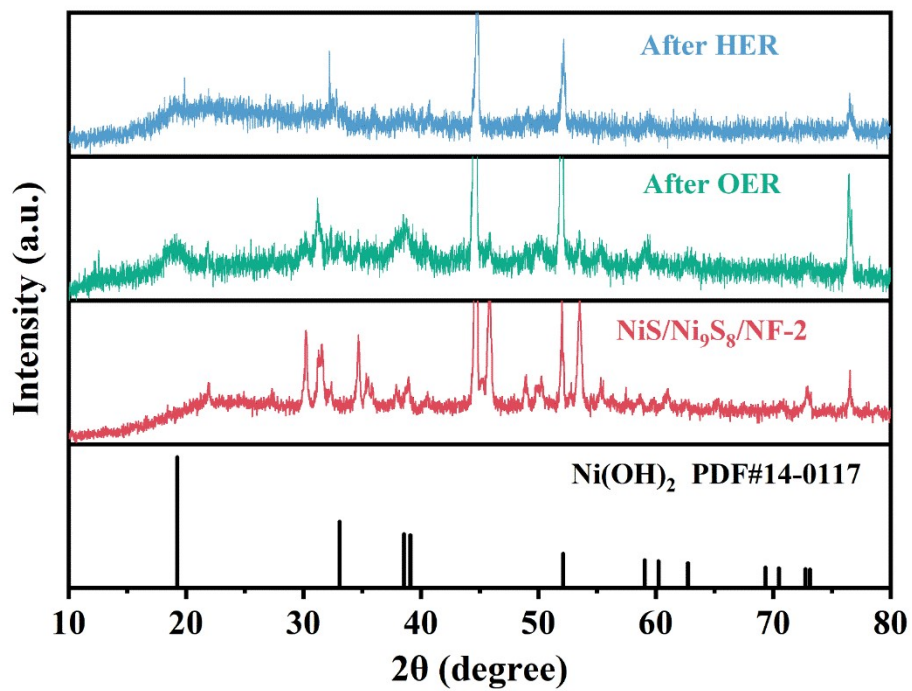


Figure S13. XRD patterns of NiS/Ni₉S₈/NF-2 after 100 h long term electrolysis for the HER and 80 h long term electrolysis for the OER.

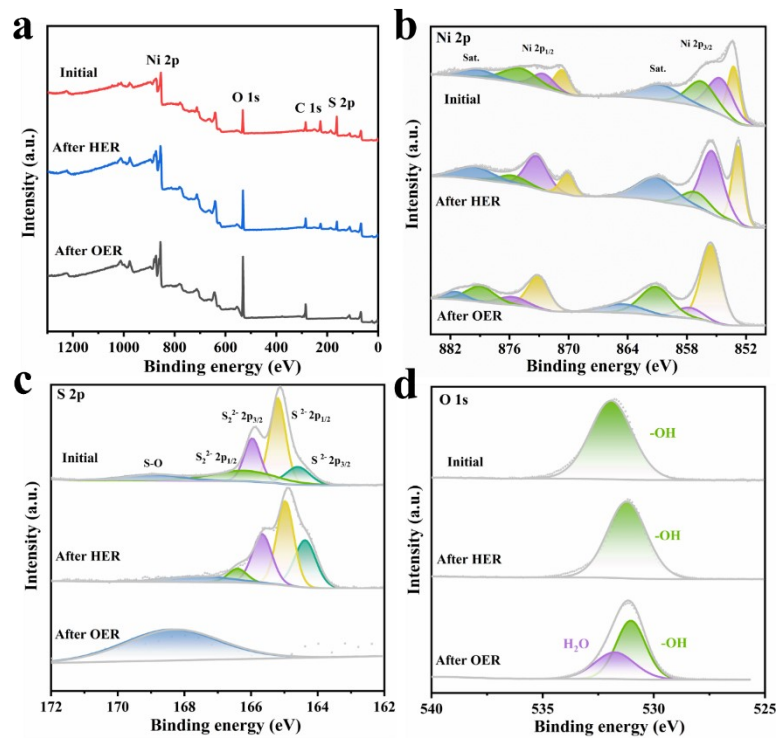


Figure S14. (a) XPS survey, (b) Ni 2p, (c) S 2p, (d) O 1s spectra of NiS/Ni₉S₈/NF-2 after i-t performance test of water splitting.

