Supporting Information

Modulation of active surface sites on Ni-Fe-S by dynamic hydrogen bubble template method for energy-saving hydrogen production

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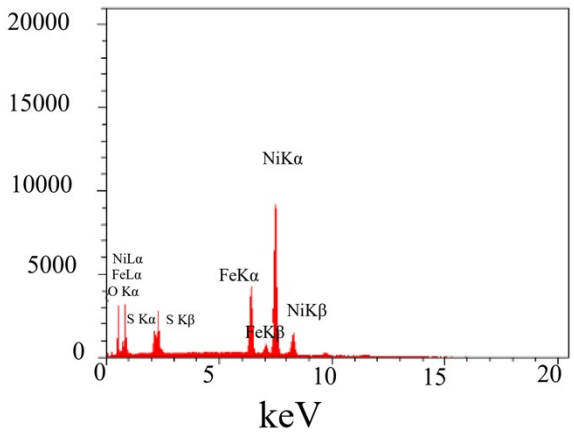


Element	Wt.%	At.%
Ni	55.28	37.76
Fe	19.77	14.20
S	11.56	14.46
O	13.39	33.58

Figure S1. EDS spectrum of optimum Ni-Fe-S 5 A.cm^{-2}/10 s/0.1 M.

Table S1. The pore diameters for different electrodes.

Catalyst	Pore Diameter (µm)
Ni-Fe-S 1 A.cm^{-2}/10 s/0.1 M	1
Ni-Fe-S 2 A.cm^{-2}/10 s/0.1 M	1 - 2
Ni-Fe-S 3 A.cm^{-2}/10 s/0.1 M	2 - 10
Ni-Fe-S 6 A.cm^{-2}/10 s/0.1 M	10 - 50
Ni-Fe-S 5 A.cm^{-2}/3 s/0.1 M	5 - 7
Ni-Fe-S 5 A.cm^{-2}/5 s/0.1 M	8 - 15
Ni-Fe-S 5 A.cm^{-2}/30 s/0.1 M	10 - 40
Ni-Fe-S 5 A.cm^{-2}/10 s/0 M	5 - 15
Ni-Fe-S 5 A.cm^{-2}/10 s/0.2 M	15 - 30
Ni-Fe-S 5 A.cm^{-2}/10 s/0.3 M	15 - 30
Ni-Fe-S 5 A.cm^{-2}/10 s/0.1 M	10 - 25

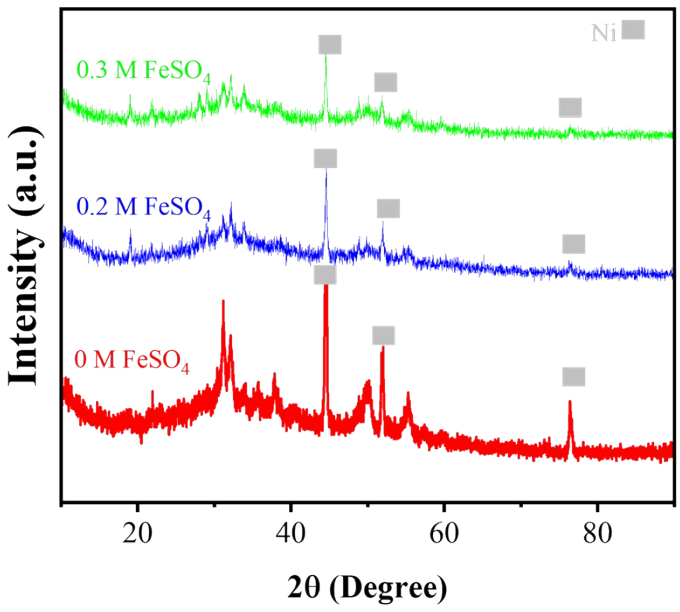


Figure S2. XRD pattern for electrodes with different concentration of $FeSO_4$.

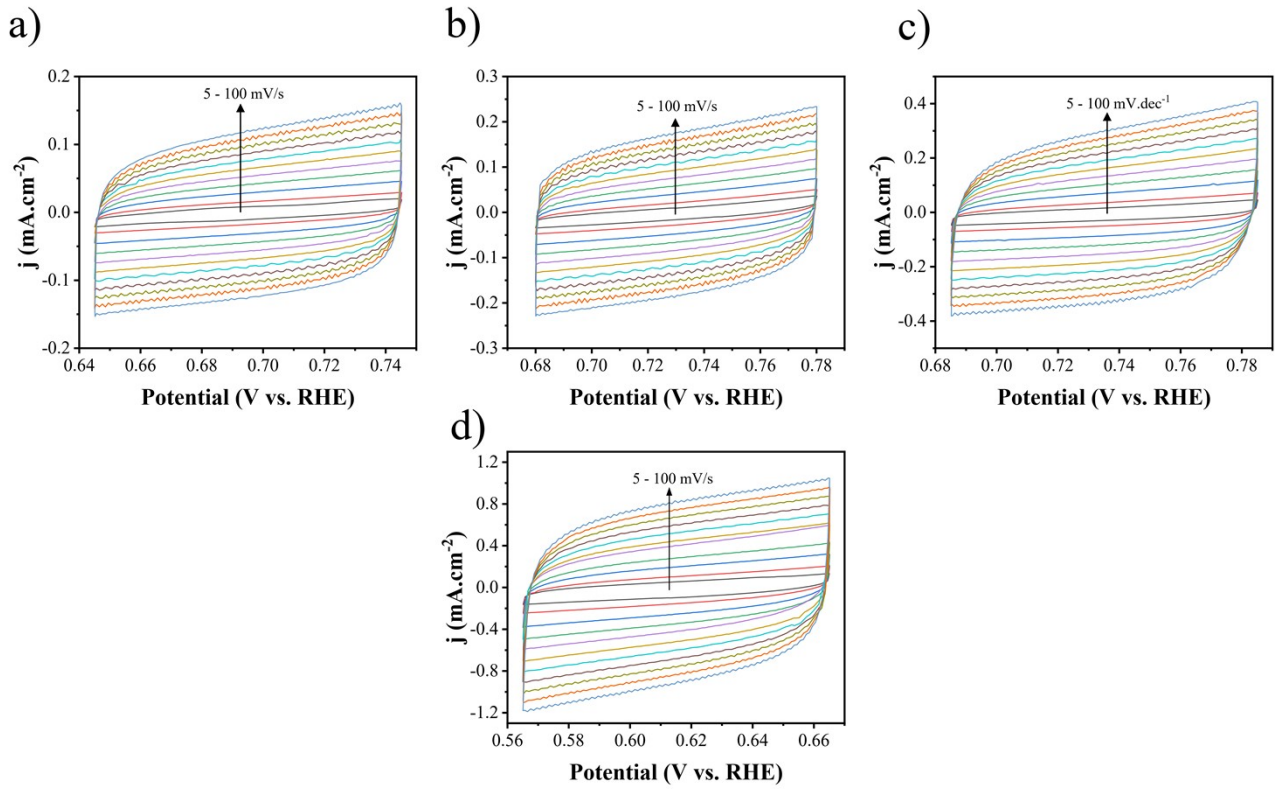


Figure S3. CV curves with different scan rates from 5-100 $mV.s^{-1}$ of samples electrodeposited with applied current density of (a) 1, (b) 2, (c) 3, (d) 6 A cm^{-2}.

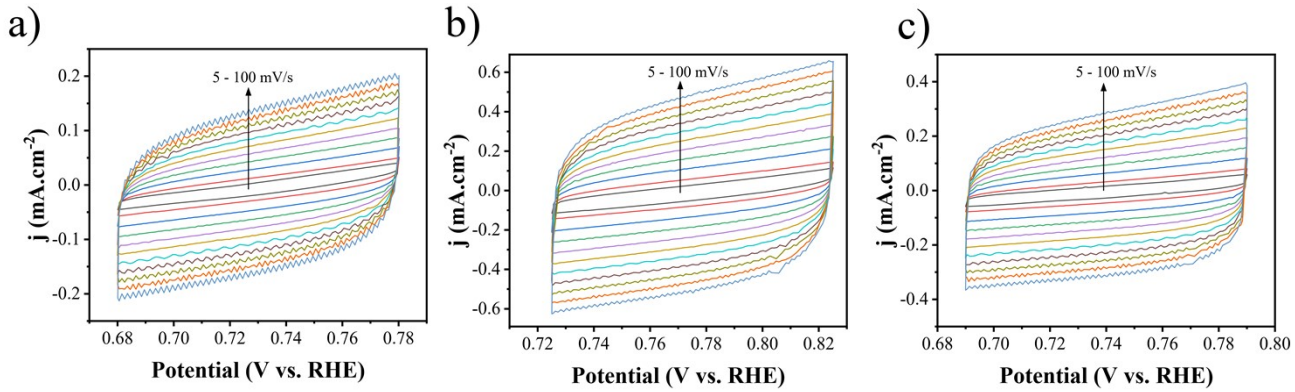


Figure S4. CV curves with different scan rates from 5-100 $mV.s^{-1}$ of samples electrodeposited with duration time of (a) 3, (b) 5, (c) 30 s.

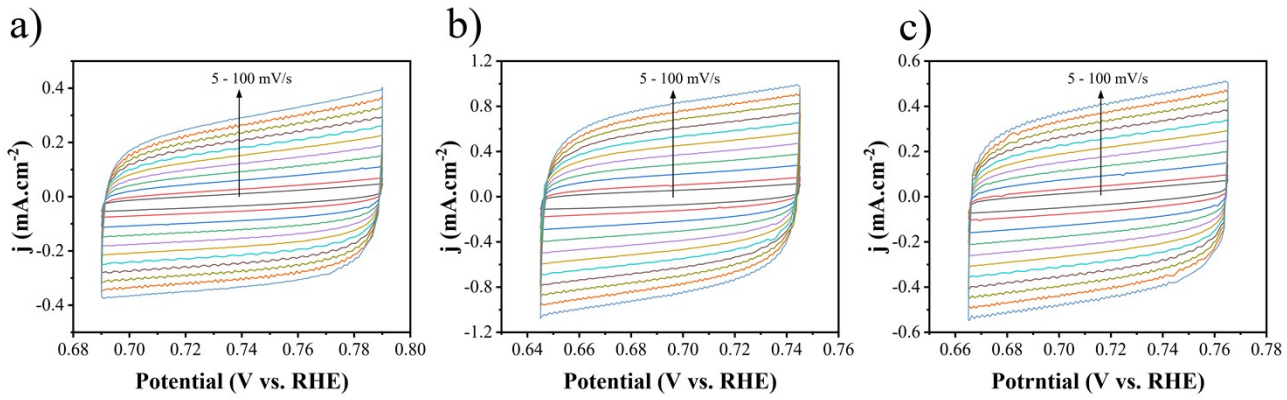
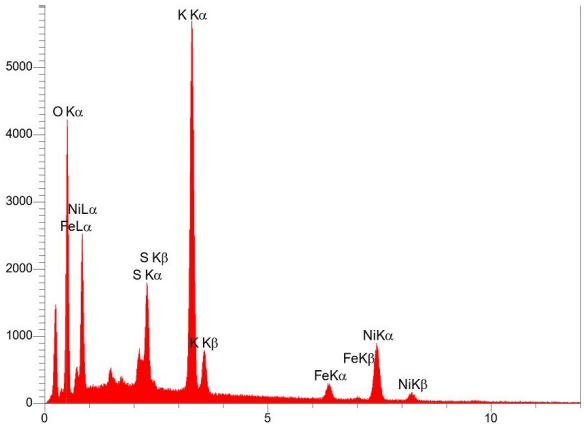


Figure S5. CV curves with different scan rates from 5-100 $mV.s^{-1}$ of samples electrodeposited with $FeSO_4$ concentration of (a) 0, (b) 0.2, (c) 0.3 M.



Element	Wt.%	At.%
O	42.47	67.80
S	5.05	4.02
K	24.16	15.78
Fe	3.60	1.65
Ni	24.72	10.75

Figure S6. EDS spectrum of optimum Ni-Fe-S 5 $A.cm^{-2}$/10 s/0.1 M after CP test.

Table S2. Comparison table of HER performances for electrocatalysts: this work vs. literatures.

Catalyst	**Electrolyte**	**η_{10} (mV vs. RHE)**	**Tafel slope ($mV.dec^{-1}$)**	**Ref.**
Ni-Fe-S	1 M KOH + 0.5 M Urea	85	50	This Study

$Ni_{0.7}Fe_{0.3}S_2$	1 M KOH	155	109	1
Ni_2Fe_2N/Ni_3Fe	1 M KOH	74	53	2
Co-Fe-Mo-S NBs	1 M KOH	300	104.13	3
FeMoS	0.5 M H_2SO_4	140	57	4
NiFeS	0.5 M H_2SO_4	81	73.1	5
NiFeMoS/NF	1 M KOH	100	121	6
$FeNi\text{-}S_x@MoS_2$	1 M KOH	155	93	7
	0.5 M H_2SO_4	110	34	
NiFe	1 M KOH	124	114	8
CoS_2 HNs	1 M KOH	215	198	9
$1T_{0.81}$- $MoS_2@Ni_2P$	1 M KOH	95	42	10
MoS_2/Ni_3S_2	1 M KOH	89	62	11
NiFeS	1 M KOH	51.4	54.3	12
NiFe/NF	1 M KOH	142	133	13
Ni-Fe-P/Cu	1 M KOH	335	63.7	14
Fe-Ni-Co	1 M KOH	134	80	15
Ni-Fe-P/NF	1 M KOH	192	142.2	16
Ni-Fe-B/NF	1 M KOH	63	56.3	17
NiFeS/C	1 M KOH	115	108	18
NiFeSe/NF	1 M KOH	117	86.5	19
MOF-Ni@MOF-Fe-S	1 M KOH + 0.5 M Urea	145	45.49	20
$Cu_2S@Ni_3Se_2$	1 M KOH + 0.5 M Urea	106	85	21
SnS/SnS_2	1 M KOH + 0.33 M Urea	170	74	22
NiCuP	1 M KOH	175	53	23
NiCo	1 M KOH	54	-	24

Table S3. Comparison table of UOR and two electrode cell performances for electrocatalysts: this work vs. literatures.

Catalyst	Electrolyte	Required potential for 10 $mA.cm^{-2}$ (V vs. RHE)	Required potential for 100 $mA.cm^{-2}$ (V vs. RHE)	V_{cell} required for 10 $mA.cm^{-2}$ (V vs. RHE)	Ref.
Ni-Fe-S	1 M KOH + 0.5 M Urea	1.26	1.3		This Study
Nanoporous NiFe	1 M KOH + 0.33 M Urea	1.33	-	1.55	25
$NiFe(OH)_x/Ni_3N$	1 M KOH + 1 M Urea	1.36	-	-	26
MOF-Ni@MOF-Fe-S	1 M KOH + 0.5 M Urea	1.34	-	1.53	20
N-C doped NiFe	1 M KOH + 1 M Urea	-	1.37	1.50	27
NiFe NSs/NF	1 M KOH + 0.33 M Urea	1.33	1.37	1.40	28
$Cu_2S@Ni_3Se_2$	1 M KOH + 0.5 M Urea	1.338	-	1.48	21
SnS/SnS_2	1 M KOH + 0.33 M Urea	-	1.39	1.36	22
Ni(OH)S nanosheets	1 M KOH + 0.33 M Urea	1.34	-	-	29
Co-Ni-S	1 M KOH + 0.33 M Urea	1.31	1.35	-	30

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Video S1: Video of Ni-Fe-S contact angle test.

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