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In situ generation Ni/Fe hydroxide layers by anodic etching of Ni/Fe

film for efficient oxygen evolution reaction

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Figure S1. Effect of glycerol (Gly) in electrolyte studied by CV in 0.1 M KOH (scan rate of 20 mV s⁻¹).



Figure S2. (a) CV curves and (b) Tafel plots of the different concentration of NH_4F in 0.1 M KOH (scan rate of 20 mV s⁻¹).



Figure S3. (a) CV curves and (b) Tafel plots of the different concentration of H₂O in 0.1 M KOH (scan rate of 20 mV

s⁻¹).



Figure S4. Effect of (a) temperature and (b) potential value during the anodization process studied by CV in 0.1 M KOH (scan rate of 20 mV s⁻¹).



Figure S5. EDS results of fluorinated Ni/Fe (before CV treatment) and Ni/Fe hydroxide layers (after CV treatment).



Figure S6. CV curves of the different anodizing time of NiFe alloy in 0.1 M KOH (scan rate of 20 mV s⁻¹).



Figure S7. XPS results of surface atomic% of fluorinated Ni/Fe (before CV treatment) and Ni/Fe hydroxide layers (after CV treatment).



Figure S8. LSV polarization curves of Ni (the grey), Fe (the red) and Ni/Fe (the blue) under anodization and CV treatment for OER electrocatalytic activity in 0.1 M KOH.



Figure S9. LSV polarization curves of Ni/Fe hydroxide and fluorinated Ni/Fe for OER electrocatalytic activity in 0.1 M KOH.



Figure S10. LSV polarization curves of Ni/Fe hydroxide (the black) and IrO_2 (loading mass: 0.90 mg cm⁻² (the blue), 0.28 mg cm⁻² (the red)) for OER electrocatalytic activity.



Figure S11. Effect of different Ni hydroxide/Fe hydroxide ratio for OER electrocatalytic activity studied by LSV in 0.1 M KOH.



Figure S12. Capacitive CV of (a) NiFe alloy, (b) fluorinated Ni/Fe and (c) Ni/Fe hydroxide recorded at different scan rates, where no apparent faradaic process has taken place.

Catalyst	Electrolyte	η (mV)	Tafel slop	Ref.
			(mV dec ⁻¹)	
Ni/Fe hydroxide	0.1 M KOH	240	47	This work
NiHCF	0.1 M KOH	364	50	[1]
Ni/Fe-150	0.1 M KOH	341	68	[1]
Ni/Fe-300	0.1 M KOH	310	44	[1]
Ni/Fe-450	0.1 M KOH	342	57	[1]
Ni/Fe-600	0.1 M KOH	370	69	[1]
$Sr_{2}Fe_{1.3}Ni_{0.2}Mo_{0.5}O_{6\cdot\delta}$	0.1 M KOH	360	59	[2]
NiFe-SW	1 M KOH	220	38.9	[3]
Ni–Fe LDH DSNCs	1 M KOH	272	71	[4]
NiFeO _x (OH) _y	1 M KOH	250	39	[5]
FeOOH/LDH	1 M KOH	174	46	[6]

Table S1. Selected summary of NiFe-based OER catalyst.

Table S2. XPS results of the Ni/Fe ratio with different samples.

Complex	Atomic (%)		
samples –	Ni	Fe	
Ni: Fe=1:1, CV potential 0~0.8V	55.42	44.58	
Ni: Fe=3:1, CV potential 0~0.8V	89.53	10.47	
Ni: Fe=1:1, CV potential 0~0.4V	59.14	40.86	

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