Supporting Information

$NiSe_2/FeSe_2$ nanodendrite: A highly efficient electrocatalyst for oxygen evolution reaction

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Experimental section

Chemicals and Materials. Oleylamine (OAm) and octadecene (ODE) are purchased from aladin. Nickel aceylacetonate and iron aceylacetonate was bought from changcheng chemicals, selenium powder is purchased from Tianjin kermel chemical. Potassium hydroxide, hexane, isopropyl alcohol and ethanol are purchased from Sinopharm Chemical Reagent Ltd. All chemical reagents are used in the experiments directly without further treatment.

Synthesis of $Ni_xFe_{1-x}Se_2$

A mixture of 0.1 mmol of $Ni(acac)_2$, 0.1mmol of $Fe(acac)_2$ and 0.4 mmol selenium powder was dissolved in the mixed solution of 8 ml of OAm and 2 ml of ODE. The temperature of solution was raised to 120 ^oC in a nitrogen flow to dissolve metal precursors. Moisture and oxygen was removed through evacuation at 120 ^oC with an

oil pump for 20 min. Then the system was refilled with nitrogen and the mixture solution was heated to 280 ^{o}C. After keeping at 280 ^{o}C for 60 min, the solution was cooled to room temperature in air. The synthesized $Ni_{0.5}Fe_{0.5}Se_2$ catalyst was collected by centrifugation and washed with hexane and ethanol for three times. The obtained black powder was dried under vacuum and stored in nitrogen atmosphere for further use.

Other $Ni_xFe_{1-x}Se_2$ (x=0.25, 0.75) catalysts were prepared in the similar method except that the metal precursors ratio were varied. For example, $Ni_{0.25}Fe_{0.75}Se_2$ was synthesized with 0.05 mmol of $Ni(acac)_2$, 0.15 mmol of $Fe(acac)_2$ and 0.4 mmol selenium, $Ni_{0.75}Fe_{0.25}Se_2$ was produced using 0.15 mmol of $Ni(acac)_2$, 0.05 mmol of $Fe(acac)_2$ and 0.4 mmol selenium as precursors. The preparing process of $NiSe_2$ and $FeSe_2$ was the same as $Ni_xFe_{1-x}Se_2$ in the absence of $Fe(acac)_2$ or $Ni(acac)_2$.

Characteristic

X-ray power diffraction patterns were measured on a Bruker D8 Advance X-ray diffractometer with Cu Kα radiation (λ = 0.154178 nm). Transmission electron microscopy (TEM) and Energy-dispersive X-ray (EDX) characterizations were done on a Tecnai G20 U-Twin electron microscope equipped with EDX detector at an acceleration voltage of 200 kV. Fe doped $NiSe_2$ catalysts was dissolved in hexane through sonication. The sample of TEM was prepared by casting a drop of Fe doped $NiSe_2$ solution on a carbon coated Cu grid. X-ray photoelectron spectroscopy (XPS) measurement was performed with a Thermo Fischer ESCALAB 250Xi spectrophotometer. Inductively coupled plasma-atomic emission spectroscopy (ICP-

AES) was conducted on IRIS Intrepid II XSP.

Ink preparation

10 mg of $Ni_xFe_{1-x}Se_2$ was mixed with the equal mass of XC-72 in 20 mL of hexane and stirred for 12 h in a nitrogen flow. The $Ni_xFe_{1-x}Se_2$/XC-72 was collected by centrifugation and washed by hexane. It was dried under vacuum at room temperature and stored in inert atmosphere for next use. For preparing catalyst ink, 5 mg of catalyst was dissolved in 1 mL Nafion/ isopropyl alcohol (0.1% Nafion) with sonication for 1 h to ensure that catalyst was dispread in solution uniformly.

Electrochemical measurement

The test for OER was performed on an electrochemical station (CHI 760E) with a rotating disc electrode (RDE) in a three electrode configuration. 18 μL of catalyst ink was deposed on the surface (surface area = 0.196 cm^2) of a glass carbon electrode (GC) and dried on air to vapor the solvent. The mass catalyst loading of all catalysts is ~0.45 mg/cm^2. The GC electrode covered with $Ni_xFe_{1-x}Se_2$/XC-72 served as working electrode and a platinum foil and a mercuric oxide electrode (MOE) immersed in 1 M KOH were used as the counter electrode (CE) and reference electrode (RE), respectively. OER measurements were conducted in 1 M KOH solution at room temperature. The potential of Hg/HgO/OH- electrode versus a reversible hydrogen electrode (RHE) was measured in saturated hydrogen atmosphere with 20 wt % Pt/C as electrocatalyst to calibrate the potential of the system. In 1 M KOH, the equation E

(RHE) = E (MOE) + 0.92 is considered. Cyclic voltammetry (CV) measurements were measured at a scan rate of 5 mV/s from 1.23 to 1.7 V vs RHE with the rotating rate of 1600 rpm after activating catalysts through a quick scan rete of 500 mV/s until reaching a stable state. We calculate the electrochemically active surface area (ECSA) by measuring double-layer capacitance in nitrogen saturated 1 M KOH solution through CVs at a scan rate of 10, 20, 30, 40, 50 mV/s. Generally specific capacitance of C_s=0.04 mF/cm^2 is used to divide double-layer capacitance.[18-20]

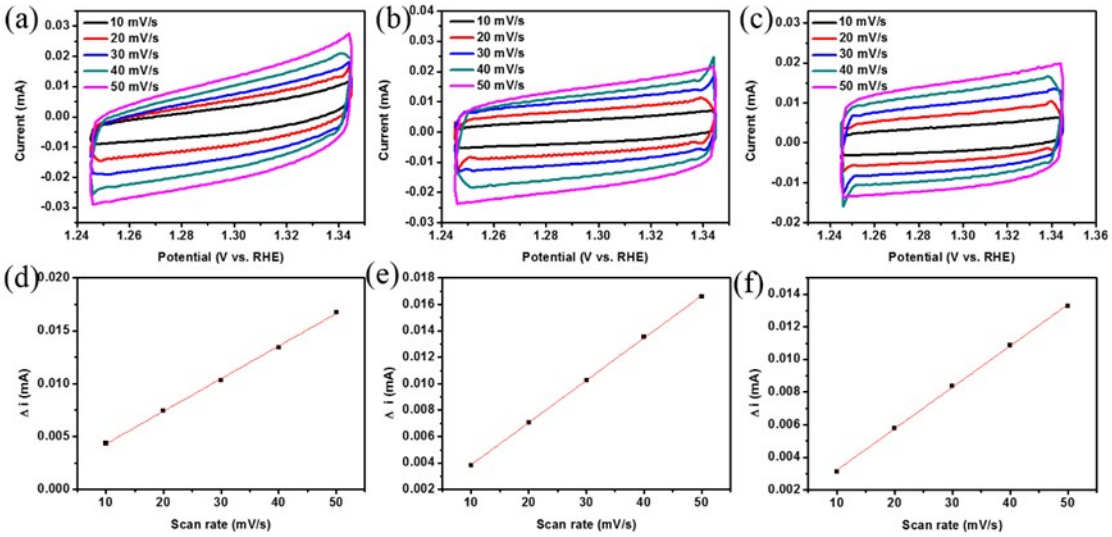

Figure S1 Electrochemical double layer capacitance curves on $Ni_{0.75}Fe_{0.25}Se_2$ (a), $Ni_{0.5}Fe_{0.5}Se_2$ (b), $Ni_{0.25}Fe_{0.75}Se_2$ (c) with different scan rates from 50 mV s^{-1} to 10 mV s^{-1} in 1 M KOH. Cdl Plots of $Ni_{0.75}Fe_{0.25}Se_2$ (d), $Ni_{0.5}Fe_{0.5}Se_2$ (e), $Ni_{0.25}Fe_{0.75}Se_2$ (f).

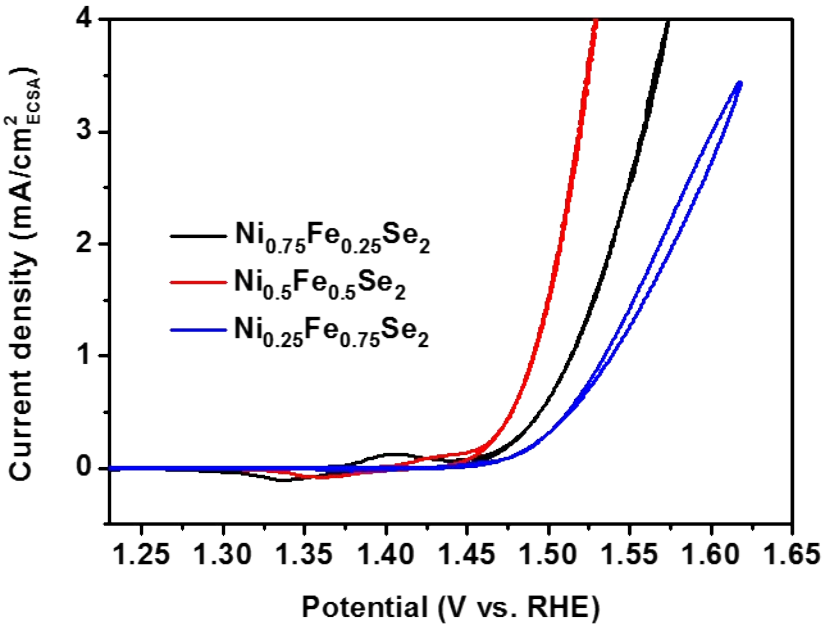

Figure S2 Specific activities normalized by ECSA of these three samples.

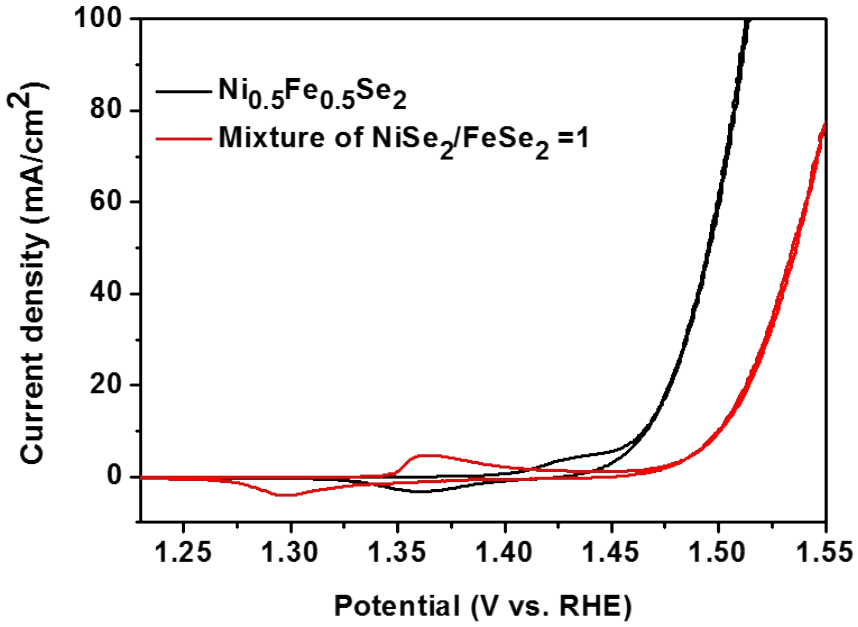

Figure S3. Polarization curves of $Ni_{0.5}Fe_{0.5}Se_2$ and physical mixture of $NiSe_2$ and $FeSe_2$.

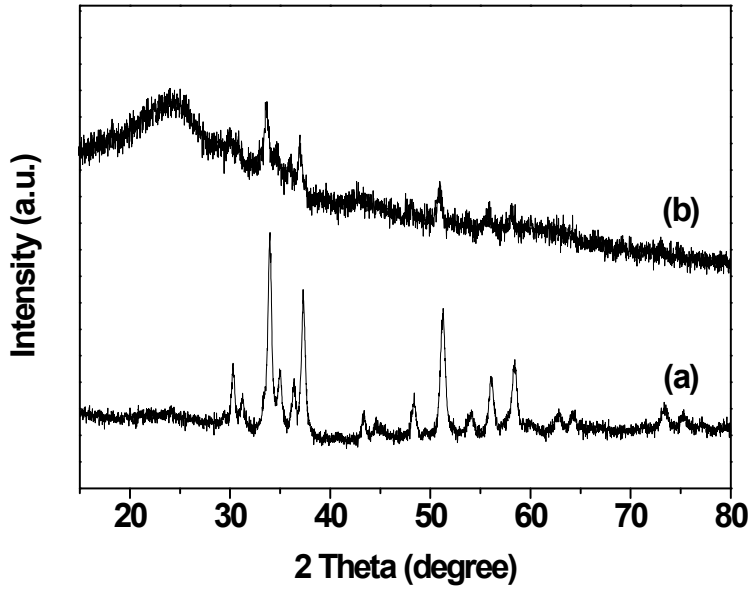

Figure S4. XRD patterns of $Ni_{0.5}Fe_{0.5}Se_2$ before (a) and after (b) stability test.

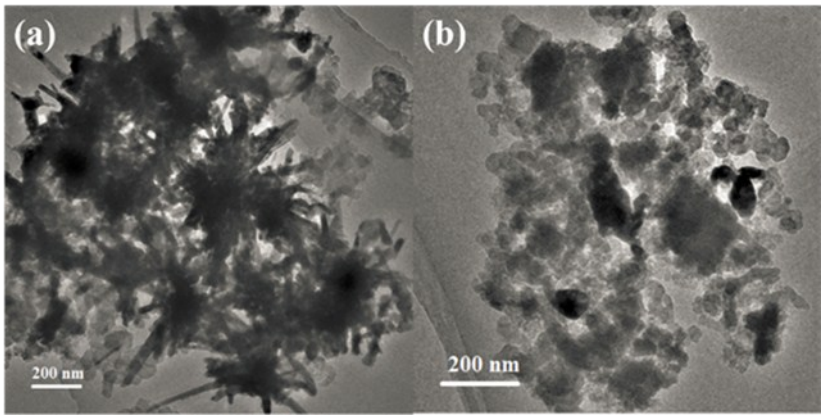

Figure S5. TEM images of $Ni_{0.5}Fe_{0.5}Se_2$/XC-72 before (a) and after (b) stability test

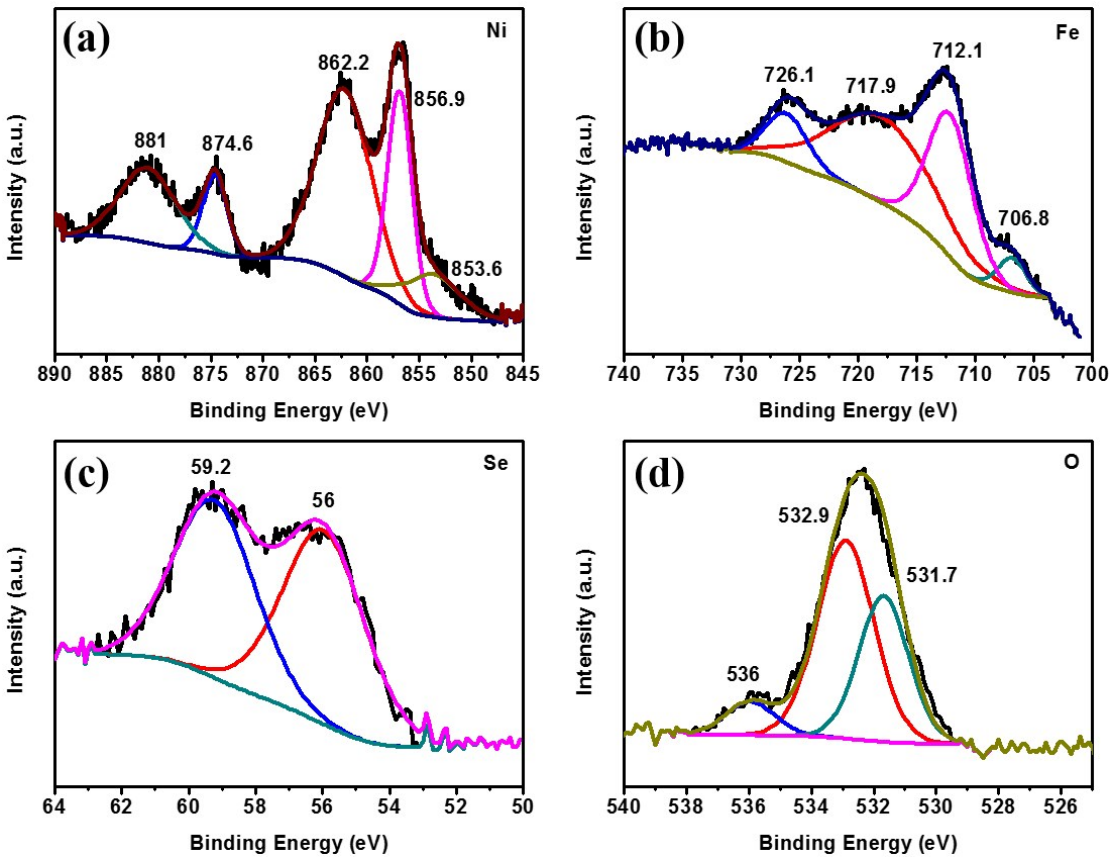

Figure S6. XPS spectra of Ni 2p (a), Fe 2p (b), Se 3d (c) and O 1s (d) in $Ni_{0.5}Fe_{0.5}Se_2$ after stability test.

Table S1 Composition of $Ni_xFe_{1-x}Se_2$ catalysts

Catalysts	1	2	3
Precursor ratio	$Ni_{0.75}Fe_{0.25}Se_2$	$Ni_{0.5}Fe_{0.5}Se_2$	$Ni_{0.25}Fe_{0.75}Se_2$
ICP result	$Ni_{0.82}Fe_{0.22}Se_2$	$Ni_{0.53}Fe_{0.47}Se_2$	$Ni_{0.27}Fe_{0.77}Se_2$

Table S2 Comparison of OER performance for $Ni_{0.5}Fe_{0.5}Se_2$ with other non-noble metal electrocatalysts towards OER in alkaline media

Catalyst	Mass loading (mg/cm^2)	Electrolyte	Tafel slop (mV/dec)	η@10mA/cm^2 mV	Ref.
Ni_3Se_2	0.217	0.3 M KOH	97.2	290	1
NiSe		1 M KOH	54	320	2
$NiSe_2$	1	1 M KOH	38	250	3
Co_7Se_8		1 M KOH	32.6	290	4
CoSe	0.28	1 M KOH	40	295	5
$CoSe_2$	0.142	0.1 M KOH	44	320	6
$CeO_2/CoSe_2$	0.2	0.1 M KOH	44	288	7
$(Ni,Co)_{0.85}Se$ @NiCo LDH	6	1 M KOH	77	216	8
CoMnP	0.284	1 M KOH	61	330	9
$NiCo_2O_4$		1 M NaOH	53	290	10
$FeSe_2$	0.01	1 M KOH	48.1	330	11
Co_9S_8	0.24	1 M KOH	50.7	294	12
Co_2B-NG	0.21	0.1 M KOH	45	360	13
FeCoO	0.36	0.1 M KOH	36.8	308	14

NiSe/NiO	4.25	1 M KOH	128	243	15
$Fe_{2-x}Mn_xP$	0.284	1 M KOH	39	440	16
CoP_2	0.285	1 M KOH	96	300	17
$Ni_{0.5}Fe_{0.5}Se_2$	0.45	1 M KOH	34.7	235	this work

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