1	Supplementary Information
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3	The combined effects of the hetero-interface of NiSe ₂ -Ni ₃ Se ₄ and Fe doping markedly boost the
4	electrocatalytic overall water splitting performance of self-supported nanosheets array electrode
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29 1. Chemicals and reagents

30 All chemical reagents were utilized without further purification. Nickel Chloride Hexahydrate 31 (NiCl₂·6H₂O, AR, 99%, Beijing Inokai Science & Technology Co., Ltd.), Ferric Chloride 32 Hexahydrate (FeCl₃·6H₂O, AR, 99%, Tianjin Zhiyuan Chemical Reagent Co., Ltd.), Terephthalic Acid (C₈H₆O₄, AR, 99%, Beijing Inokai Science & Technology Co., Ltd.), Selenium Powder (Se, AR, 33 34 99.9%, Shanghai Aladdin Biochemical Technology Co., Ltd.), N, N-dimethylformamide (C₃H₇NO, 35 AR, 99%, Tianjin Xinbute Chemical Co., Ltd.), potassium hydroxide (KOH, AR, 85%, Tianjin Zhiyuan Chemical Reagent Co., Ltd.), commercial Pt-C (20 wt%, Suzhou Sinero Technology Co., 36 Ltd.) and RuO₂ (99.5%, Ru ≥75wt%, Suzhou Sinero Technology Co., Ltd.) 37

38 2. Materials characterization

39 The morphology and detailed micro-nano structure of the as-prepared samples were characterized 40 by scanning electron microscopy (SEM) with Hitachi S-4800, transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) with FEI Talos F200X. X-ray diffraction (XRD) patterns 41 were recorded on a Bruker D8 advance X-ray diffractometer with Cu K α radiation source (λ = 42 1.54178 Å). X-ray photoelectron spectroscopy (XPS) was performed using Thermo Fisher Scientific 43 44 Escalab 250 Xi with monochromatic Al Ka at 15 kW. X-ray absorption fine structure (XAFS) spectroscopy was carried out using the Rapid XAFS 2M (Anhui Absorption Spectroscopy Analysis 45 Instrument Co., Ltd.) by transmission mode at 20 kV and 20 mA, and the Ge (620) spherically bent 46 47 crystal analyzer with a radius of curvature of 500 mm. Nitrogen (N₂) adsorption-desorption isotherms 48 were measured for specific surface area of all the prepared catalysts by using Brunauer-Emmett-Teller 49 (BET, Micromeritics ASAP 2460, USA) and corresponding Barrett-Joyner-Halenda (BJH) method 50 was used for pore size and distribution analysis. Contact angles (CAs) in both liquid and air phases 51 were measured using a contact angle-measuring instrument (Dataphysics OCA 25) under ambient 52 conditions (room temperature, 1 atm), including water droplets in air and air bubbles in aqueous media. 53 Raman spectroscopy was performed on a Bruker Senterra microscope-confocal Raman spectrometer 54 at the excitation wavelength of 532 nm.

55 3. Electrochemical measurement

56 A three-electrode system (CHI 760E, CH Instruments, China) was used to evaluate the 57 electrochemical performance of HER/OER. The samples were directly tested as the working electrode 58 (totally immersed geometric area: 50×50 mm) and the graphite rod and Hg/HgO electrode served as 59 counter and reference electrode, respectively. For comparison, 4.12 mg of Pt-C or RuO₂ was dispersed into a mixed solution containing 990 uL of anhydrous ethanol and 10 uL of Nafion solution (Du Pont, 60 61 5 wt%) and then subjected to ultrasonic treatment to prepare a homogeneous ink. Subsequently, 1000 62 μ L of the ink solution was dropped onto the surface of NF (50 × 50 mm) and dried naturally at room 63 temperature.

The working electrode was scanned by cyclic voltammetry (CV) until the signal was stabilized and then the data were collected. The linear scanning voltammetry (LSV) curves were carried out at a scan rate of 5 mV s⁻¹ in 1.0 M KOH and was corrected with 100% iR compensation. The potentials were calibrated to a reversible hydrogen electrode (RHE) according to the equation:

68
$$E (RHE) = E (Hg/HgO) + 0.098 + 0.059 \text{ pH} - 100\% \text{ iR}$$
 (1)

69 To assess the reaction kinetics, Tafel slopes were extracted from the Tafel equation:

$$\eta = b \log j + a \tag{2}$$

71 b is the Tafel slope and j denotes the current density

The electrochemical active surface area (ECSA) was studied by using the electrochemical
double-layer capacitance (C_{dl}). The ECSA was calculated:

74

$$ECSA = C_{dl}/C_s$$
(3)

75 $C_s = 0.04 \text{ mF cm}^{-2}$

76 The double layer capacitance (C_{dl}) was determined by CV curves in the non-faradic potential region
77 with different scan rates (20, 40, 60, 80 and 100 mV s⁻¹).¹

78 The electrochemical impedance spectroscopy (EIS) measurement was conducted over a 79 frequency range of 0.01-10⁵ Hz with a 5 mV AC potential perturbation. For OWS, a two-electrode 80 configuration was adopted, and the electrolyte of 1.0 M KOH was utilized.

For OWS, the assembled dual electrode device based on Fe-NiSe₂-Ni₃Se₄/NF-2 in 1.0 M KOH.

82 The stability was evaluated via CV for 2000 cycles with a scan rate of 100 mV s^{-1} and the

83 amperometric i-t method.

84 The Faraday efficiencies for HER/OER were calculated based on the ratio of the volume of 85 actual (V_{actual}) H₂/O₂ evolved to the theoretical one ($V_{theoretical}$):

86 Faraday efficiency =
$$\frac{V_{actual}}{V_{theoretical}} \times 100\%$$
 (4)

87 The actual volumes of generated H_2/O_2 gas were recorded using the drainage method. The theoretical 88 volume of generated H_2/O_2 can be calculated using the formula:

89
$$V_{\text{theoretical}} = \frac{It V_{\text{m}}}{zF}$$
(5)

90 where I is current (A), t is time (s), V_m is molar volume of H₂/O₂ gas (23.6 L mol⁻¹, 298 K, 100 kPa in
91 Shuimogou District, Urumqi, Xinjiang), F is the Faraday constant (96485 C mol⁻¹), z is electron
92 number transferred per molecule (z is 2 and 4 for HER and OER, respectively).

93 4. Theoretical calculations

94 Spin-polarized calculations were performed using density functional theory (DFT) methods implemented in the Vienna ab initio simulation package (VASP).^{2, 3} The generalized gradient 95 96 approximation (GGA) with the Perdew-Burke-Ernzerhof (PBE)was employed to account for electron exchange and correlation interactions.⁴ Additionally, the DFT-D3 method of Grimme was utilized to 97 98 consider Van der Waals interactions between the adsorbed intermediate and the substrate.^{5, 6} Projected 99 augmented wave (PAW) potentials were selected to describe ionic cores and valence electrons using a plane wave basis set with a kinetic energy cutoff of 450 eV.7, 8 Geometry optimization was iterated 100 until reaching a total energy tolerance converged to 10⁻⁵ eV and changes in atomic forces were less 101 than 0.02 eV Å⁻¹. The Brillouin zone was sampled on Monkhorst-Pack meshes of $1 \times 1 \times 1$ for 102 structural optimization and electronic structure calculations.⁹ Finally, a vacuum layer of 15 Å was 103 104 added to the surface to eliminate artificial interactions between periodic images.

105 The Gibbs free energy of the intermediates during HER and OER processes was determined as106 (equation 2):

$$\Delta G = E_{\rm ads} + \Delta E_{\rm ZPE} - T\Delta S \tag{4}$$

108 where * denotes an active site, E_{ads} represents the adsorption energy of the intermediate.¹⁰ ΔE_{ZPE} and 109 ΔS denote the change in zero-point energy and entropy between absorbed and gas states, respectively, 110 with *T* set at 300 K. The E_{ZPE} and *S* values for the adsorbates were obtained from vibrational 111 frequencies calculations using harmonic approximation, neglecting contributions from the slabs. The 112 thermodynamic numerical values mentioned above were obtained from the standard database in the 113 National Institute of Standards and Technology (NIST) web book.¹¹

114 In the process of HER, the adsorption energy of *H (E_{*H}) was determined using the following 115 formula (equation 3):

116
$$E_{*\mathrm{H}} = \Delta G_{*\mathrm{H}} - (\Delta E_{\mathrm{ZPE}} - T\Delta S_{*\mathrm{H}})$$
(5)

117 According to the free energy analysis method developed by Nørskov et al, the calculated value of 118 ($\Delta E_{ZPE} - T\Delta S_{*H}$) is approximately 0.24 eV.¹² Therefore, the aforementioned equation was formulated 119 as follows (equation 4):

120

$$E_{*\rm H} = \Delta G_{*\rm H} - 0.24 \tag{6}$$

121 The calculation results were presented in Table S2, accompanied by the corresponding122 calculation models depicted in Figure S6.

According to equation 5, the thermodynamic potential for OER is determined to be 1.23 V under standard conditions (T = 298.15 K, P = 1 bar, pH = 0) (equation 5).

125 $2H_2O \rightarrow O_2 + 4H^+ + 4e^-$ (7)

126 And the elementary steps for OER under alkaline conditions are outlined as follows (equations 6-9) [].

$$* + OH^{-} \rightarrow *OH + e^{-}$$
(8)

$$*OH + OH^{-} \rightarrow *O + H_2O + e^{-}$$
(9)

$$*O + OH^{-} \rightarrow *OOH + e^{-}$$
(10)

130
$$*OOH + OH^- \rightarrow O_2 + H_2O + e^-$$
 (11)

131 The total sum of ΔG_i (i = 1-4) is specifically equivalent to the inverse value of twice the experimental

132 Gibbs free energy of forming one water molecule (ΔG^{exp}_{H2O}) (equation 10).¹³

133
$$-2\Delta G^{\exp}_{\text{H2O}} = -nE = (-4e) \times (-1.23 \text{ V}) = 4.92 \text{ eV}$$
(12)

134 in which *n* refers to the number of electrons involved in the reaction, and *E* represents the voltage. In

135 this case, calculating bond energy in O_2 is avoided which is hard to obtain an accurately value within 136 GGA-DFT.¹⁴⁻¹⁶ Then energy changes (ΔG_i (i=1-4)) of simulated each step in the OER process under 137 alkaline conditions are presented as follows (equations 11-14):

$$\Delta G_1 = \Delta G_{*\rm OH} - eU + \Delta G_{\rm H+}(\rm pH)$$
(13)

139
$$\Delta G_2 = \Delta G_{*O} - \Delta G_{*OH} - eU + \Delta G_{H+}(pH)$$
(14)

140
$$\Delta G_3 = \Delta G_{*OOH} - \Delta G_{*O} - eU + \Delta G_{H+}(pH)$$
(15)

141
$$\Delta G_4 = 4.92 - \Delta G_{*OOH} - eU + \Delta G_{H+}(pH)$$
(16)

142 where U is the applied potential.^{17, 18} The free energy change of the protons relative to the above 143 specified electrode at non-zero pH is represented by Nernst equation as equation 15,

144
$$\Delta G_{\mathrm{H}+}(\mathrm{pH}) = -k_B T \ln(10) \times \mathrm{pH}$$
(17)

145 The calculation results were presented in Table S3, accompanied by the corresponding146 calculation models depicted in Figure S7.

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148

150 FeNi-BDC/NF-x (x = 1, 2, 3) and Ni-BDC/NF

- 152 water and drop-cast the suspension onto single-crystal silicon wafers, thereby ensuring the collected
- 153 signals originated solely from the target compounds.
- 154

¹⁵¹ To eliminate this substrate interference, the samples was ultrasonically exfoliated in deionized

- 156 Figure S2. SEM images of (a, b) Ni-BDC/NF, (c, d) FeNi-BDC/NF-1, (e, f) FeNi-BDC/NF-2 and (g, h)
- 157 FeNi-BDC/NF-3 at different magnifications

159 Figure S3. The full-scan XPS spectrum of Fe-NiSe₂-Ni₃Se₄/NF-2

160

- 162 Figure S4. (a) Fe K-edge XANES spectra and (b) FT-EXAFS spectra at Fe K-edge for Fe-NiSe₂.
- 163 Ni₃Se₄/NF-2 and reference samples; (c) FT-EXAFS fitting curve at Fe K-edge for Fe-NiSe₂-
- 164 Ni₃Se₄/NF-2 in R-space; WT-EXAFS spectra at Fe K-edge for (d) Fe foil, (e) FeO, and (f) Fe-NiSe₂-
- 165 Ni₃Se₄/NF-2

- 167 Figure S5. N₂ adsorption-desorption isotherms and corresponding pore size distribuiton of (a) NiSe₂-
- 168 Ni₃Se₄/NF, (b) Fe-NiSe₂-Ni₃Se₄/NF-1, (c) Fe-NiSe₂-Ni₃Se₄/NF-2, and (d) Fe-NiSe₂-Ni₃Se₄/NF-3

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- 171 Figure S6. An optimized model of (a) NiSe₂-Ni₃Se₄/NF, *H intermediates absorbed on the Se sites (b)
- 172 or Ni sites (c) at the NiSe₂-Ni₃Se₄ interface; An optimized model of (d) Fe-NiSe₂-Ni₃Se₄/NF-2, *H
- 173 intermediates absorbed on the Fe sites (e), Se sites (f) or Ni sites (g) at the Fe-NiSe₂-Ni₃Se₄ interface
- 174

- 175
- 176 Figure S7. An initial model of (a) NiSe₂-Ni₃Se₄/NF and theoretical models of (b) *OH, (c) *O, and (d)
- 177 *OOH intermediates absorbed on the Ni sites at the NiSe₂-Ni₃Se₄ interface; An initial model of (e) Fe-
- 178 NiSe₂-Ni₃Se₄/NF-2 and theoretical models of (f) *OH, (g) *O, and (h) *OOH intermediates absorbed
- 179 on the Fe sites at the Fe-NiSe₂-Ni₃Se₄ interface; An initial model of (i) Fe-NiSe₂-Ni₃Se₄ and theoretical
- 180 models of (j) *OH, (k) *O, and (l) *OOH intermediates absorbed on the Ni sites at the Fe-NiSe₂-
- 181 Ni_3Se_4 interface

- 183 Figure S8. (a) Summary of various NixSey-based electrocatalysts for HER at 10 mA cm⁻² in 1.0 M
- 184 KOH; CV curves at different scan rates of (b) Fe-NiSe₂-Ni₃Se₄/NF-2, (c) NiSe₂-Ni₃Se₄/NF, (d) Fe-
- 185 $NiSe_2-Ni_3Se_4/NF-1$, (e) Fe-NiSe_2-Ni_3Se_4/NF-3 and (f) FeNi-BDC/NF-2 for HER

187	Figure S9. (a)	Summary of various	NixSey-based	electrocatalysts for	or OER at 10 mA	. cm ⁻² in 1.0 M
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188 KOH; CV curves at different scan rates of (b) Fe-NiSe₂-Ni₃Se₄/NF-2, (c) NiSe₂-Ni₃Se₄/NF, (d) Fe-

 $NiSe_2-Ni_3Se_4/NF-1$, (e) Fe-NiSe_2-Ni_3Se_4/NF-3 and (f) FeNi-BDC/NF-2 for OER

- 195 Figure S10. The ECSA-normalized LSV curves of Fe-NiSe₂-Ni₃Se₄/NF-2 and contrast samples for (a)
- 196 HER and (b) OER
- 197

- 199 Figure S11. SEM images at different magnifications of Fe-NiSe₂-Ni₃Se₄/NF-2 after the stability test
- $200 \quad \text{for } (a,b) \text{ HER and } (c,d) \text{ OER}$

- 202 Figure S12. (a) XRD pattern of Fe-NiSe₂-Ni₃Se₄/NF-2 after the stability test for HER and OER; The
- 203 high-resolution XPS spectra of (b) Fe 2p, (c) Ni 2p and (d) Se 3d for Fe-NiSe₂-Ni₃Se₄/NF-2 after the
- **204** stability test of HER and OER

206 Figure S13. The in-situ Raman spectra for Fe-NiSe₂-Ni₃Se₄/NF-2 under HER condition

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208	Figure S14. The in-situ Raman spectra for (a) NiSe ₂ -Ni ₃ Se ₄ /NF and (b) Fe-NiSe ₂ -Ni ₃ Se ₄ /NF-2 under
210	OER condition
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Sample	Shell	CN	R(Å)	$\sigma^2(Å^2)$	$\Delta E_0(eV)$	R fator
Fe K-edge ($S_0^2 = 0.63$)						
Fe Foil	Fe-Fe1	8.0	2.46±0.02	0 0043+0 0024	5 312+1 765	0.0071
10101	Fe-Fe2	6.0	2.85±0.02	0.0015±0.0021	5.512±1.705	
Fe-NiSe2-Ni2Se4/NF-2	Fe-Se1	1.5±0.7	2.32±0.02	0.0035+0.0012	3 827+1 396	0.0060
	Fe-Se2	5.5±0.3	2.42±0.04	0.0035±0.0012	5.027±1.570	

216 Table S1 EXAFS fitting parameters at the Fe K-edge for Fe-NiSe $_2$ -Ni₃Se $_4$ /NF-2 and Fe foil

218 Table S2. Specific Gibbs free energy (ΔG) of intermediates during the alkaline HER process

Electrocatalyst	Electrocatalyst Active sites at Intermediate		ΔG (eV)
NiSe2-Ni2Se4/NF	Ni	Ni *H	
	Se	*Н	0.61
	Fe	*Н	0.84
Fe-NiSe ₂ -Ni ₃ Se ₄ /NF-2	Ni	*Н	0.38
	Se	*Н	0.29

219 Note, * represents a Co active site on the surface.

Electrocatalyst	Active sites at interface	Intermediate	U = 0 eV	U = 1.23 eV
			ΔG (eV)	
	Ni	*OH	0.84	-0.38
NiSe2-Ni2Se4/NF		*0	0.73	-0.50
		*OOH	2.39	1.15
		O ₂	0.96	-0.27
	Fe	*OH	1.09	-0.14
		*0	0.95	-0.28
		*OOH	2.24	1.01
Fe-NiSea-NiaSea/NF-2		O ₂	0.64	-0.59
	Ni	*OH	0.64	-0.59
		*0	1.45	0.22
		*OOH	1.82	0.58
		O ₂	1.01	-0.21

231 Table S3. Specific Gibbs free energy (ΔG) of intermediates during the alkaline OER process

232 Note, * represents a Co active site on the surface.

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