Supporting Information

One-step growth of nitrogen-decorated iron-nickel sulfide nanosheets for oxygen evolution reaction

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Experimental Section

Materials: Thiourea ($CS(NH_2)_2$, AR) and urea ($CO(NH_2)_2$, AR) were purchased from Tianjin Damao Chemical Reagent Factory. Sulfur (S, AR) was purchased from Aladdin Chemical Reagent Factory. Ni foams, NiFe alloy foams (Ni:85%; Fe:15%) and NiFe alloy foams (Ni:50%; Fe:50%) were purchased from Kunshan Jiayisheng Electronics Co., Ltd. Hydrochloric acid (HCl solution, 36-38%, AR) and potassium hydroxide (KOH, AR) were purchased from Guangzhou Chemical Reagent Factory. The deionized water was purified by the RO-DI system.

Synthesis of N-$(Ni,Fe)_3S_2$/NIF: 1 mmol thiourea was transferred into a corundum boat (60 mm×30 mm×15 mm). A piece of NiFe alloy foam (5.0 mm×2.0 mm×1.0 mm) after being cleaned by dilute HCl solution and deionized water was also transferred into the corundum boat. Then the corundum boat was transferred into a tube furnace. Annealing temperature is 300 °C for 1 hour. When cooled down to room temperature, N-$(Ni,Fe)_3S_2$/NIF was took out.

Synthesis of N-Ni_3S_2/NF: 1 mmol thiourea was transferred into a corundum boat (60 mm×30 mm×15 mm). A piece of Ni foam (5.0 mm×2.0 mm×1.0 mm) after being cleaned by dilute HCl solution and deionized water was also transferred into the corundum boat. Then the corundum boat was transferred into a tube furnace. Annealing temperature is 300 °C for 1 hour. When cooled down to room temperature, N-Ni_3S_2/NF was took out.

Synthesis of H:N-$(Ni,Fe)_3S_2$/NIF: 1 mmol sulfur and 1 mmol urea were transferred into a corundum boat (60 mm×30 mm×15 mm). A piece of NiFe alloy foam (5.0 mm×2.0 mm×1.0 mm) after being cleaned by dilute HCl solution and deionized water was also transferred into the corundum boat. Then the corundum boat was transferred into a tube furnace. Annealing temperature is 450 °C for 1 hour. When cooled down to room temperature, H:N-$(Ni,Fe)_3S_2$/NIF was took out.

Synthesis of N-(Ni,Fe)S/NIF*:* 1 mmol sulfur and 1 mmol urea were transferred into a corundum boat (60 mm×30 mm×15 mm). A piece of NiFe alloy foam (5.0 mm×2.0 mm×1.0 mm) after being cleaned by dilute HCl solution and deionized water was also transferred into the corundum boat. Then the corundum boat was transferred into a tube furnace. Annealing temperature is 300°C for 1 hour. When cooled down to room temperature, N-(Ni,Fe)S/NIF was took out.

Synthesis of α-NiFeS/NIF*:* 1 mmol sulfur was transferred into a corundum boat (60 mm×30 mm×15 mm). A piece of NiFe alloy foam (5.0 mm×2.0 mm×1.0 mm) after being cleaned by dilute HCl solution and deionized water was also transferred into the corundum boat. Then the corundum boat was transferred into a tube furnace. Annealing temperature is 300°C for 1 hour. When cooled down to room temperature, α-NiFeS/NIF was took out.

Synthesis of N-$(Ni,Fe)_9S_8$/NIF: 1 mmol thiourea was transferred into a corundum boat (60 mm×30 mm×15 mm). A piece of NiFe alloy foam (5.0 mm×2.0 mm×1.0 mm, Ni:50%; Fe:50%) after being cleaned by dilute HCl solution and deionized water was also transferred into the corundum boat. Then the corundum boat was transferred into a tube furnace. Annealing temperature is 300 °C

for 1 hour. When cooled down to room temperature, N-$(Ni,Fe)_3S_2$/NIF was took out.

Characterizations: TG-IR data were acquired on a TG-209/Vector^{TM}-22 combined instrument. SEM measurements were taken on a Quanta 400/INCA/HKL scanning electron microscope. XRD data were obtained on a D-MAX 2200 VPC diffractometer with Cu Ka radiation (λ=1.54056Å). TEM measurements were acquired on a JEM-2010HR electron microscopy. XPS measurements were carried out on an ESCALAB 250 spectroscopy.

Electrochemical measurements: Electrochemical measurements were tested on a Bio-logic VMP3 electrochemical analyzer. The OER electrochemical measurements were performed in a typical three-electrode device. The counter electrode is a graphite rod and the reference electrode is a reversible hydrogen electrode (RHE). Polarization curves at a scan rate of 1 mV s^{-1} were measured in O_2-saturated 1 M KOH solution at 25 °C. The chronopotentiometric was used to test stability and the OER potential at a constant current density of 10 mA cm^{-2}. All of the potentials are iR-corrected.

Table S1. Comparisons of OER performance for various non-precious electrocatalysts in alkaline solution (η_{10}: overpotential at the current density of 10 mA cm^{-2}).

Catalysts	**η_{10} (mV)**	**Electrolyte**	**Ref.**
N-$(Ni,Fe)_3S_2$/NIF	**167**	**1 M KOH**	**This work**
FeCoW oxyhydroxides	191	1 M KOH	Science 2016 , 352, 333.
$W_{0.5}Co_{0.4}Fe_{0.1}$/NF	250	1 M KOH	Angew. Chem. Int. Ed. 2017, 129, 4573.
FeCo/Co_4N	280	1 M KOH	Adv. Mater. 2017, 29, 1704091.
porous MoO_2	260	1 M KOH	Adv. Mater. 2016, 28, 3785.
FeNiP-NP	180	1 M KOH	Adv. Mater. 2017, 29, 1704075.
$NiSe_2$ nanosheets	330	1 M KOH	Adv. Mater. 2017, 29, 1701687.
Mn@$Co_xMn_{3-x}O_4$	246	1 M KOH	Adv. Mater. 2017, 29, 1701820.
Fe-Ni_3S_2 /FeNi	282	1 M KOH	Small, 2017, 13, 1604161.
NiS	320	1 M KOH	ACS Energy Lett. 2016, 1, 195.

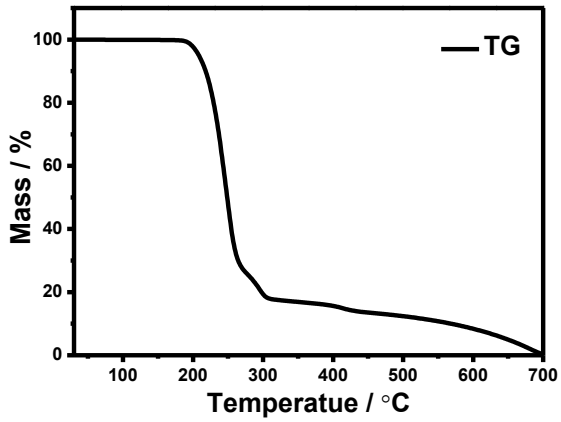

Figure S1. TG curve of thiourea during the annealing process in a nitrogen atmosphere.

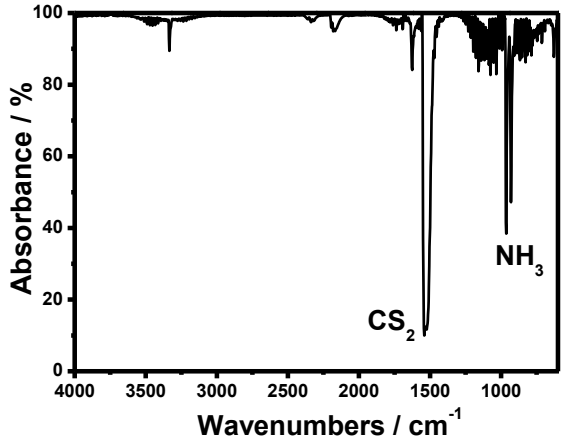

Figure S2. IR curve of decomposition products of thiourea in a nitrogen atmosphere.

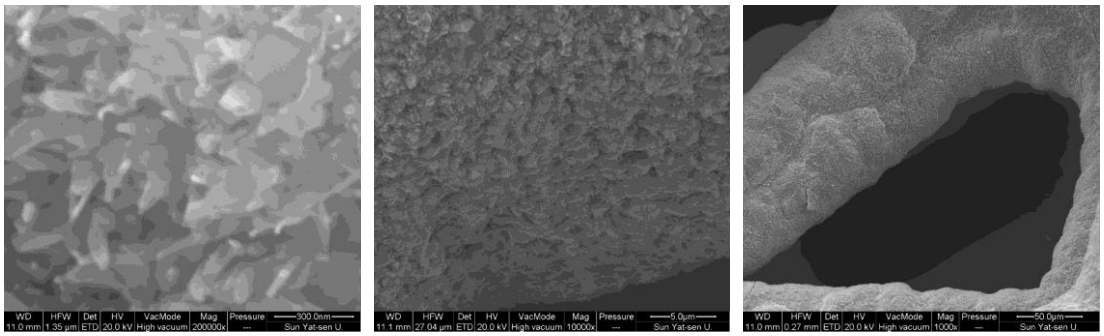

Figure S3. SEM images of N-$(Ni,Fe)_3S_2$/NIF.

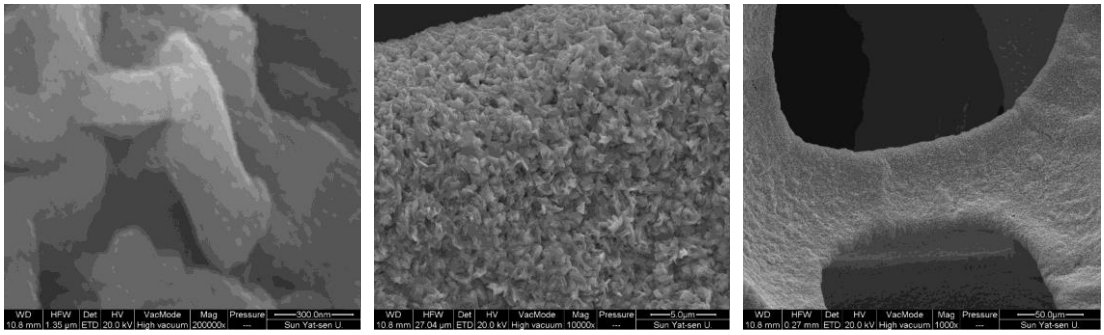

Figure S4. SEM images of N-Ni_3S_2/NF

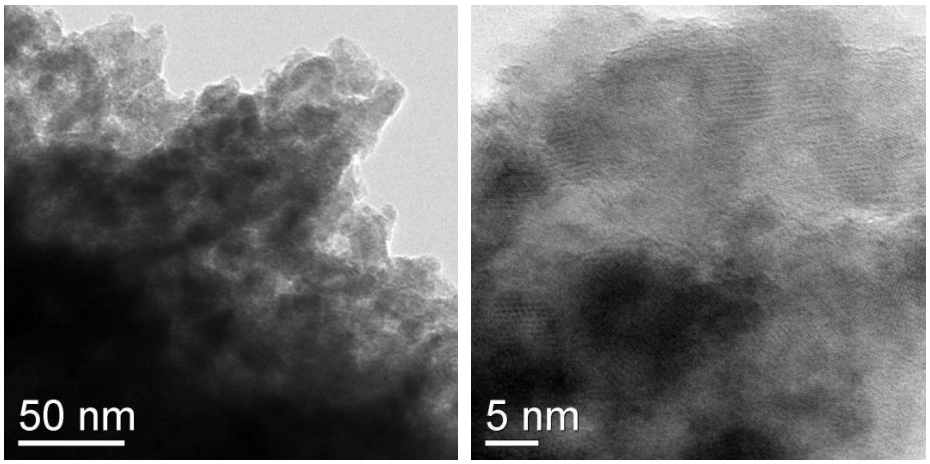

Figure S5. TEM images of N-$(Ni,Fe)_3S_2$.

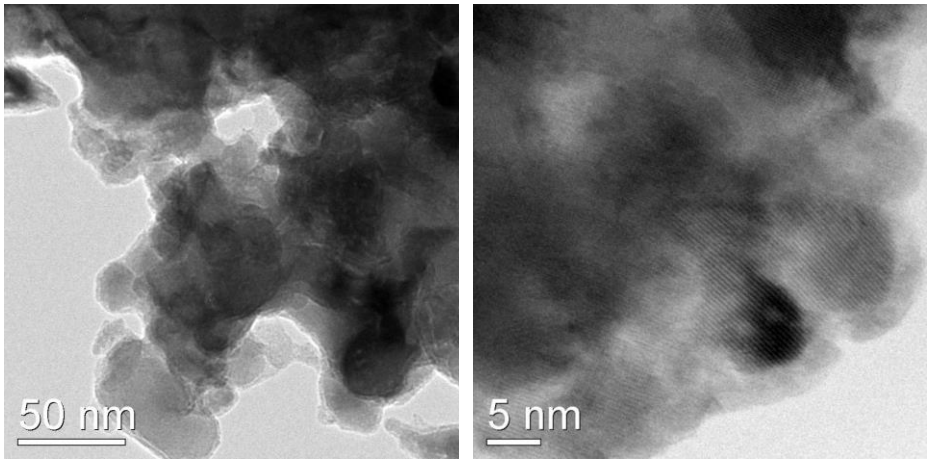

Figure S6. TEM images of N-Ni_3S_2.

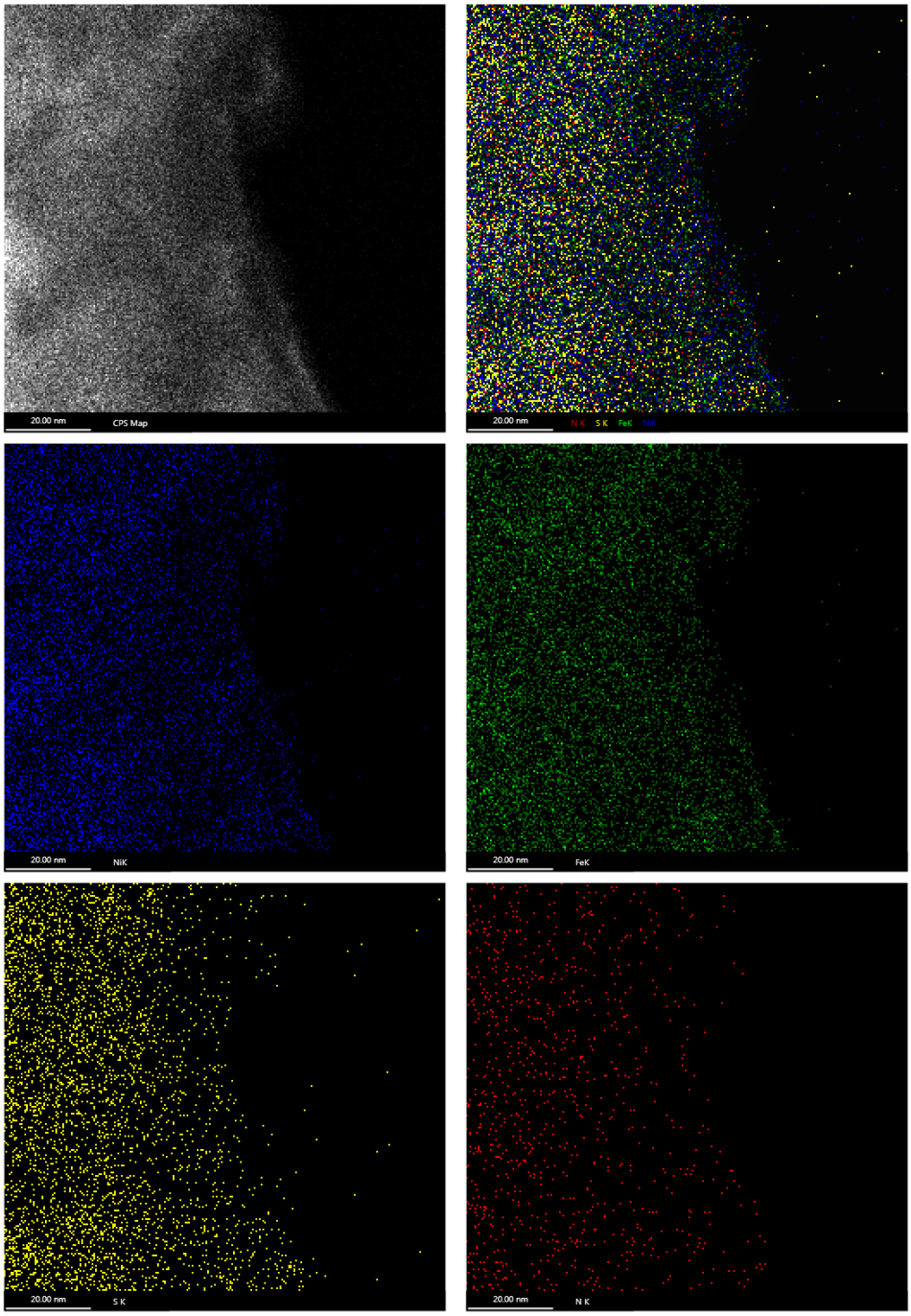

Figure S7. EDX elemetal mapping images of N-$(Ni,Fe)_3S_2$.

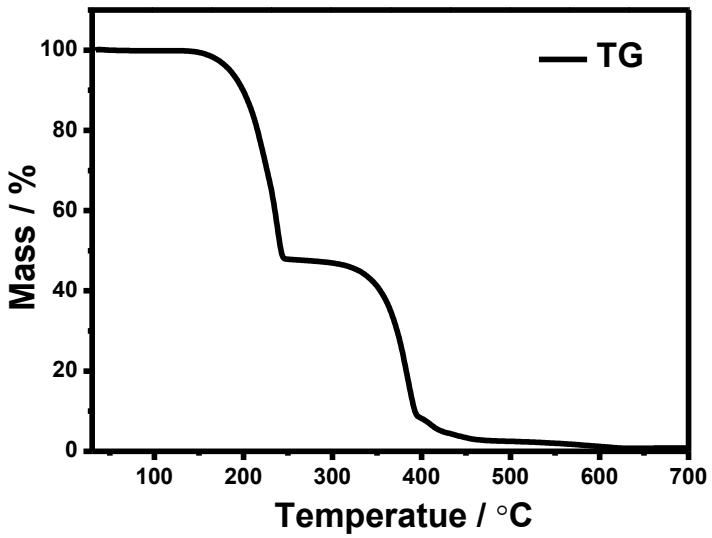

Figure S8. TG curve of urea during the annealing process in a nitrogen atmosphere.

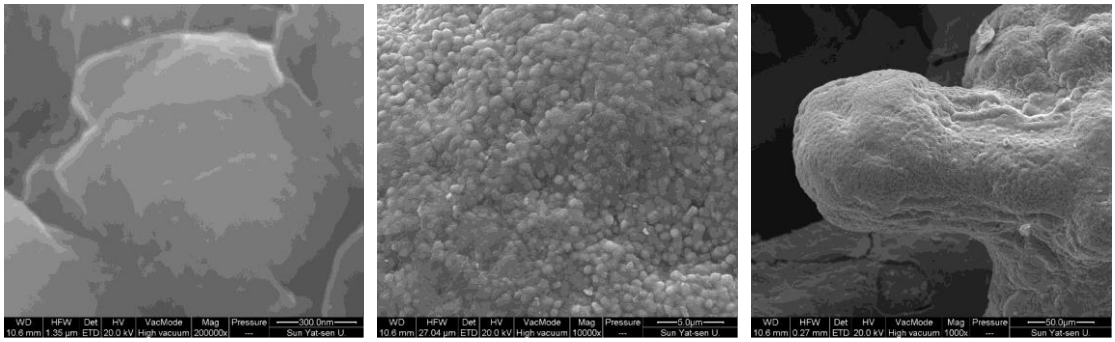

Figure S9. SEM images of H:N-$(Ni,Fe)_3S_2$/NIF.

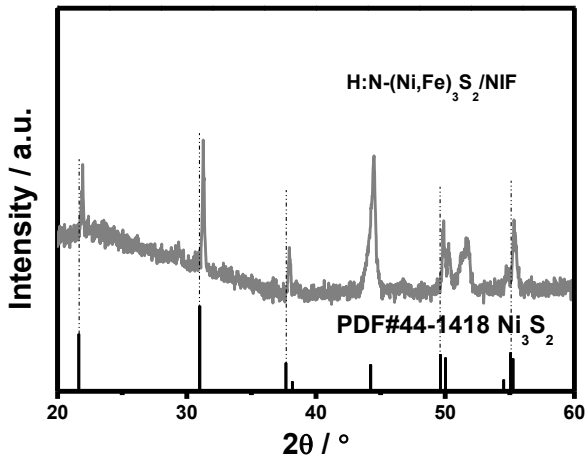

Figure S10. XRD pattern of H:N-$(Ni,Fe)_3S_2$/NIF.

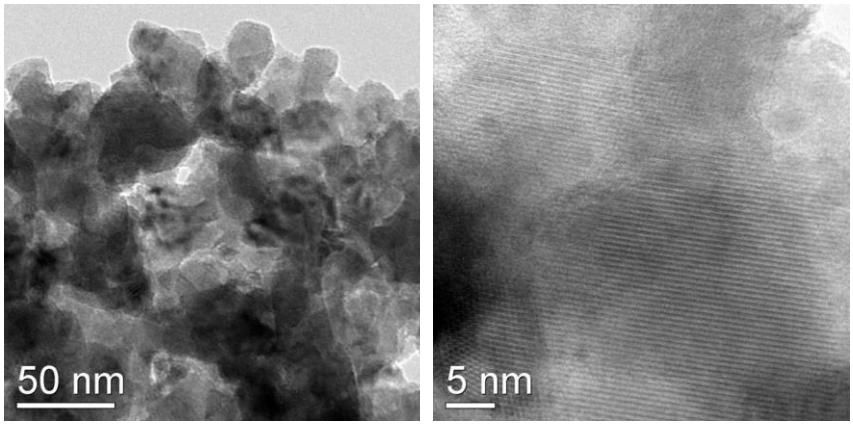

Figure S11. TEM images of H:N-$(Ni,Fe)_3S_2$.

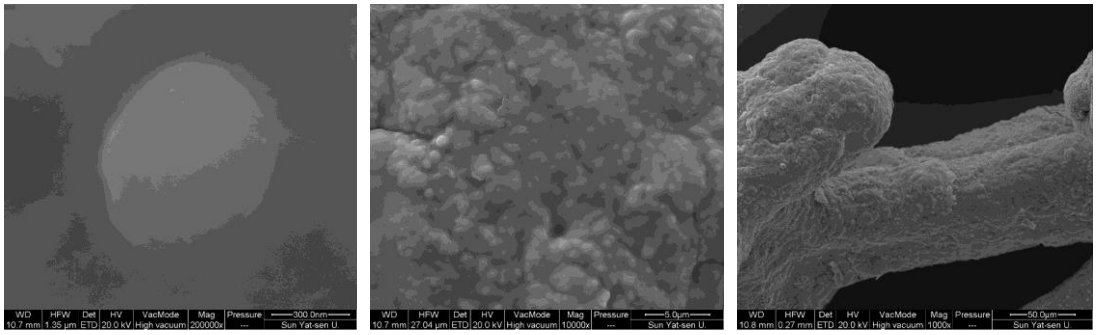

Figure S12. SEM images of N-(Ni,Fe)S/NIF.

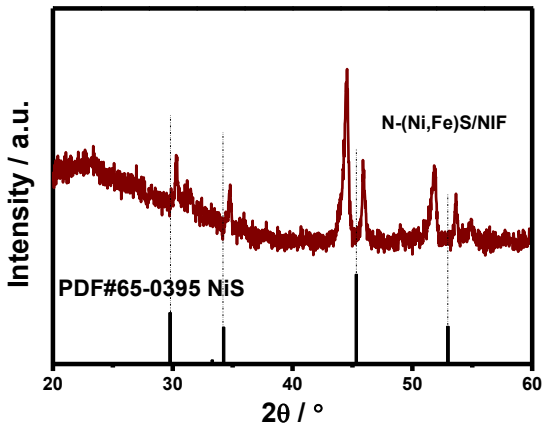

Figure S13. XRD pattern of N-(Ni,Fe)S/NIF.

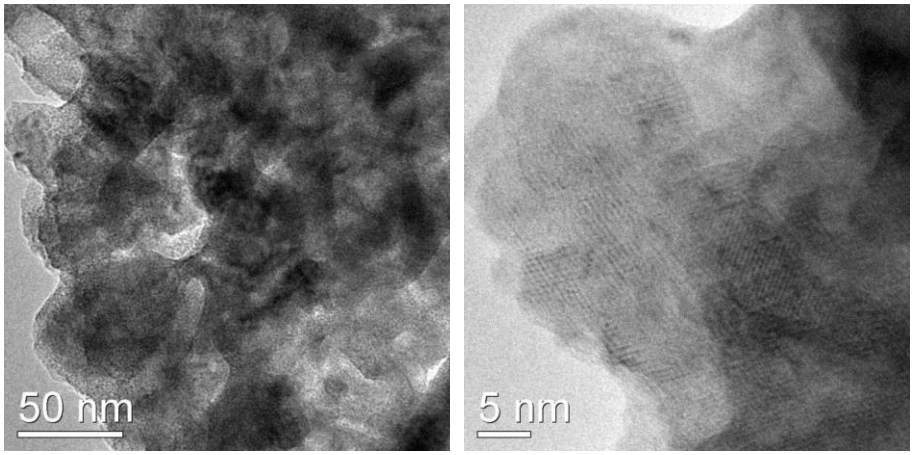

Figure S14. TEM images of N-(Ni,Fe)S.

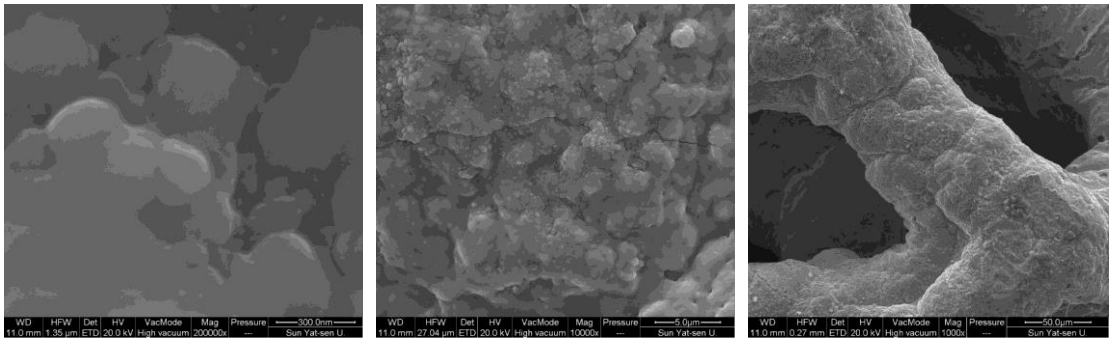

Figure S15. SEM images of α-NiFeS/NIF.

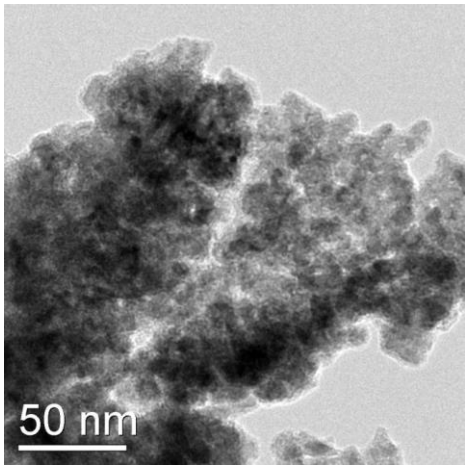

Figure S16. TEM images of α-NiFeS.

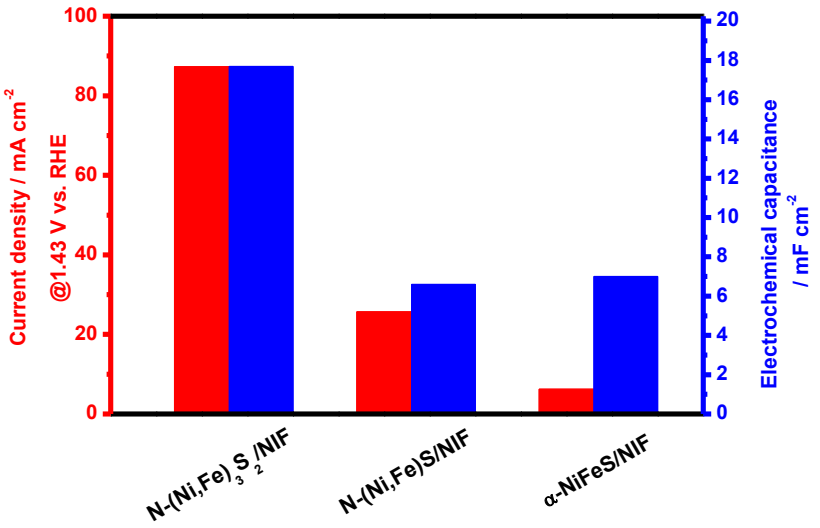

Figure S17. Current density @ 1.43 V vs. RHE and Electrochemical capacitance of N-$(Ni,Fe)_3S_2$/NIF, N-(Ni,Fe)S/NIF and α-NiFeS/NIF.

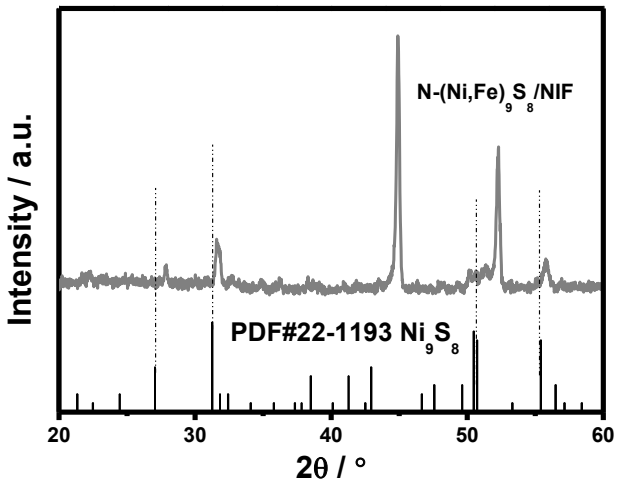

Figure S18. XRD patterns of N-$(Ni,Fe)_9S_8$/NIF(Fe:50%).

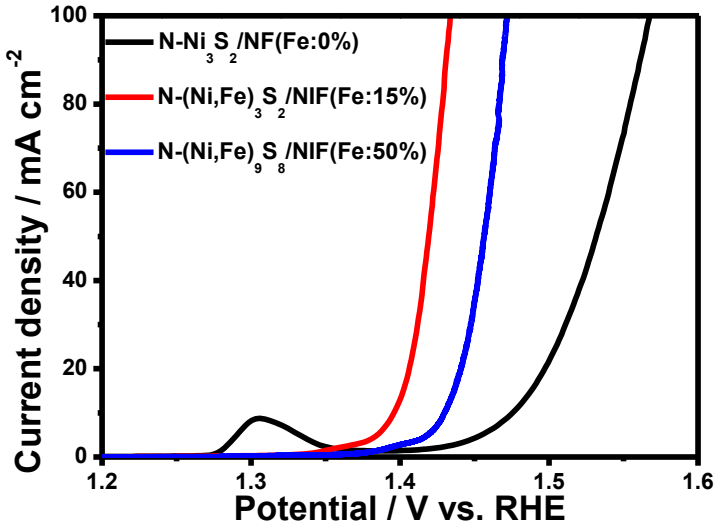

Figure S19. Polarization curves with iR-corrected of N-Ni_3S_2/NF, N-$(Ni,Fe)_3S_2$/NIF(Fe:15%) and N-$(Ni,Fe)_9S_8$/NIF(Fe:50%) in 1M KOH solution at a scan rate of 1 mV s^{-1} and 25 °C.

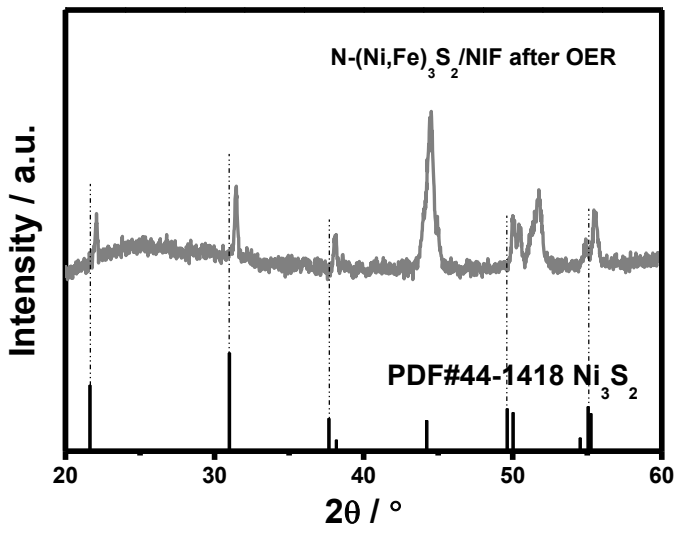

Figure S20. XRD pattern of N-$(Ni,Fe)_3S_2$/NIF after OER.

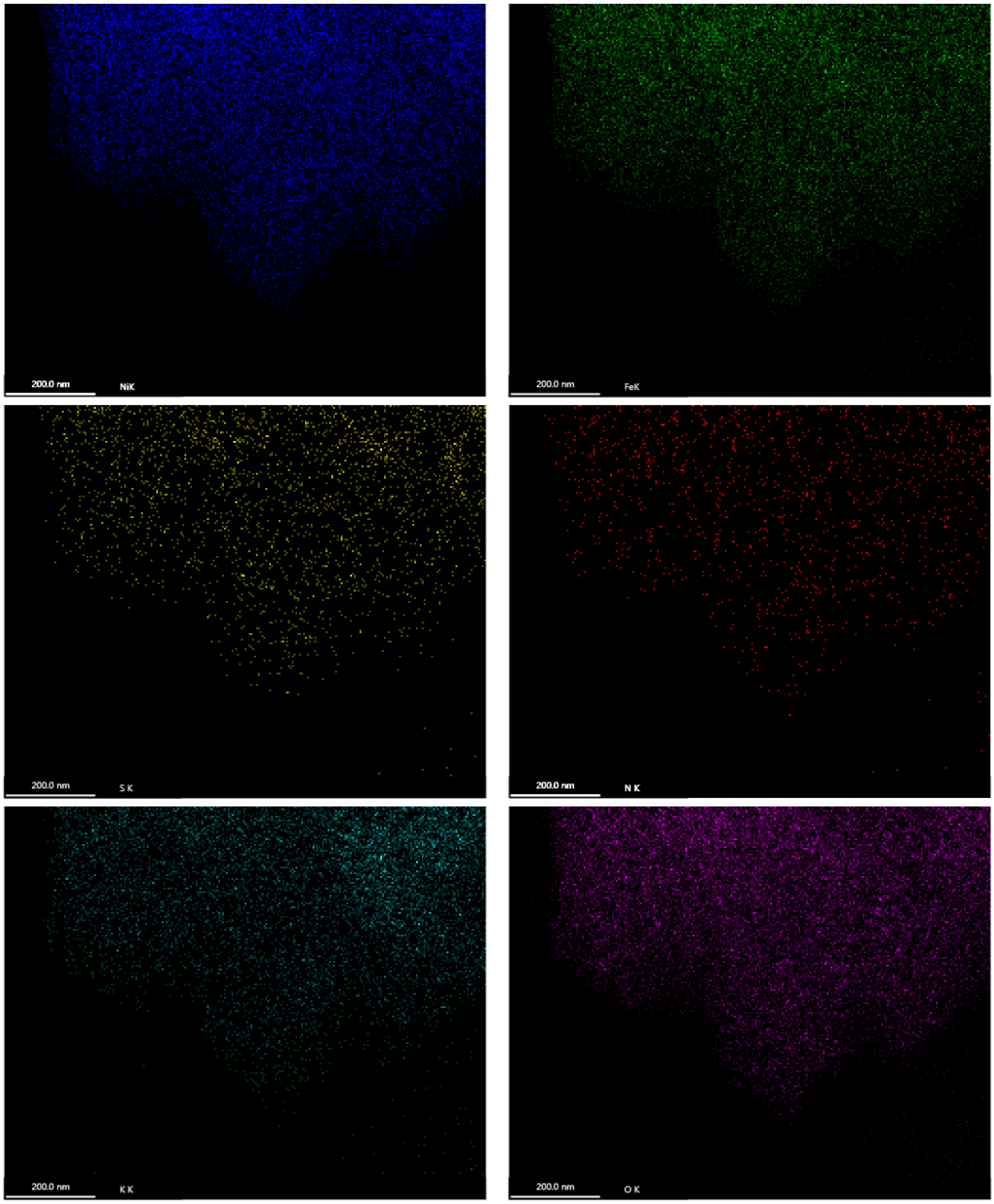

Figure S21. EDX elemetal mapping images of N-$(Ni,Fe)_3S_2$ after OER.