Electronic Supplementary Material (ESI) for New Journal of Chemistry.
This journal is © The Royal Society of Chemistry and the Centre National de la Recherche Scientifique 2023

Supporting Information

Composite structure of a-MoS_X@Ni_9S_8/NF nanoflower rods for efficient HER under thermal field

Xiaohao Jiang, Rongtao Xiao, Honggui Wang*, Ya Zhang*

School of Environmental Science and Engineering, Yangzhou University, Yangzhou 225127, China.

*Corresponding author.

E-mail: wanghg@yzu.edu.cn (H.G. Wang), zhangya@yzu.edu.cn (Y. Zhang)

Phone: +86-15358512111

Electrochemical measurement

The electrochemical tests were conducted in a 0.5 M H_2SO_4 solution (pH=0) on a CHI660D electrochemical workstation with a three-electrode system consisting of modified working electrode, a saturated calomel electrode and graphite rod. The working electrode was made up of a platinum electrode clip that holds the material. The working electrode was submerged in the electrolyte with a controlled area of 1×1 cm^2 (actual working area is 2 cm^2). IR compensation was considered in all electrochemical tests. Linear sweep voltammograms (LSV) measurements were conducted at a scan rate of 20 mV s^{-1}. Electrochemical impedance spectra (EIS) measurements were conducted at frequency from 0.1 Hz to 100 kHz at 50 mV (vs. reversible hydrogen electrode (RHE)) with an amplitude of 10 mV. Hydrogen production was obtained using Gas Chromatography (GC9790 II). All the measured potentials were converted to reversible hydrogen electrodes (RHE): $E_{(RHE)} = E_{(SCE)} + 0.0591 \times pH + 0.24$ (V).

Characterization

Morphological observations of the material were performed on a Philips Tecnai G2 F30 S-TWIN high-resolution transmission electron microscope (HRTEM) and a Hitachi S-4800 field emission scanning electron microscopy (SEM), along with high-angle annular dark field scanning TEM (HAADF-STEM) and elemental mapping. The BET surface area was carried out on the Qusdrasorb EVO Physical Sorption Apparatus. X-ray diffraction (XRD) patterns were obtained on a Bruker D8 X-ray diffractometer (Cu Kα radiation (λ = 1.5406 Å)). X-ray photoelectron spectroscopy (XPS) measurements were carried out using a Thermo Scientific ESCALAB 250Xi X-ray photoelectron spectroscopy system.

ECSA calculations and specific activity

The ECSA can be calculated from the following equation:

$ECSA=C_{dl}/C_s$

where C_{dl} is the double layer capacitance, C_s is the specific capacitance (40 μF cm^{-2}). The specific activity is obtained by normalizing the apparent current to ECSA. For the calculation of C_{dl}, cyclic voltammograms of the electrode material in the non-Faradaic region are recorded at various scan rates, a graph of scan rates versus current densities is plotted, and the slope of the graph is calculated through the linear fitting. The value of the slope is numerically equal to twice the C_{dl} value, which is therefore half the slope value.

Measurement of Faraday efficiency

Faraday efficiency can be estimated from the ratio of V_1 (the experimental recorded gas volume) to V_0 (the theoretical gas volume) during the charge transport process.

$V_0 = (1/2) \times (Q/F) \times V_m$

Faraday efficiency = V_1/V_0

where Q is the total amount of charge passed through the electrode, F is Faraday constant (96485 C mol^{-1}), V_m is the molar volume of gas (24.5 L mol^{-1}, 298 K, 101 KPa).

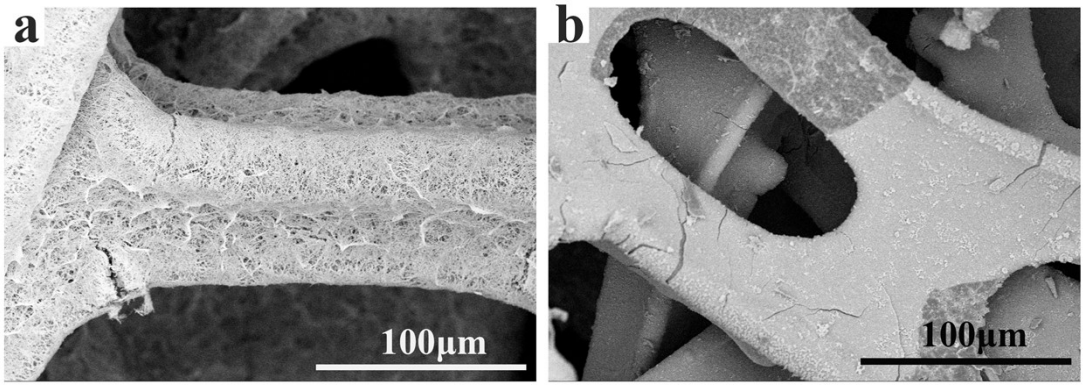

Fig. S1. SEM images of (a) Ni_9S_8/NF, (b) a-MoS_X@NF.

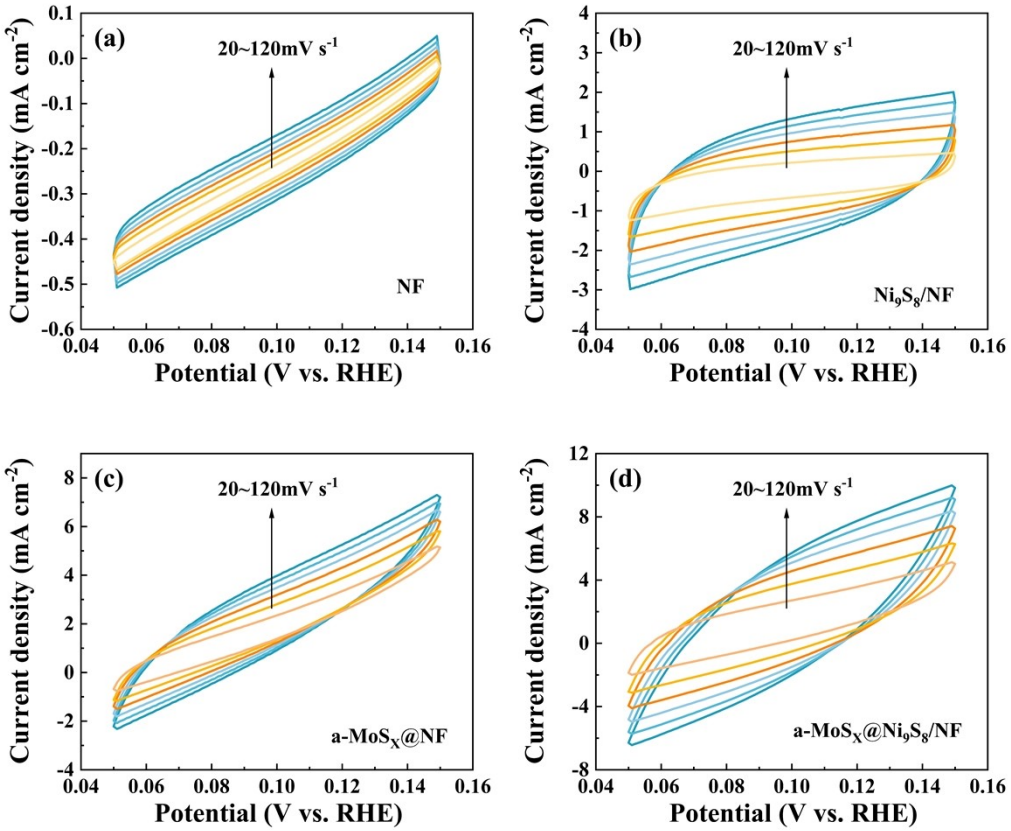

Fig. S2. (a)-(d) CV curves of NF, Ni_9S_8/NF, a-MoS_X@NF and a-MoS_X@Ni_9S_8/NF with different scan rates from 20 to 120 mV s^{-1} in 0.5 M H_2SO_4.

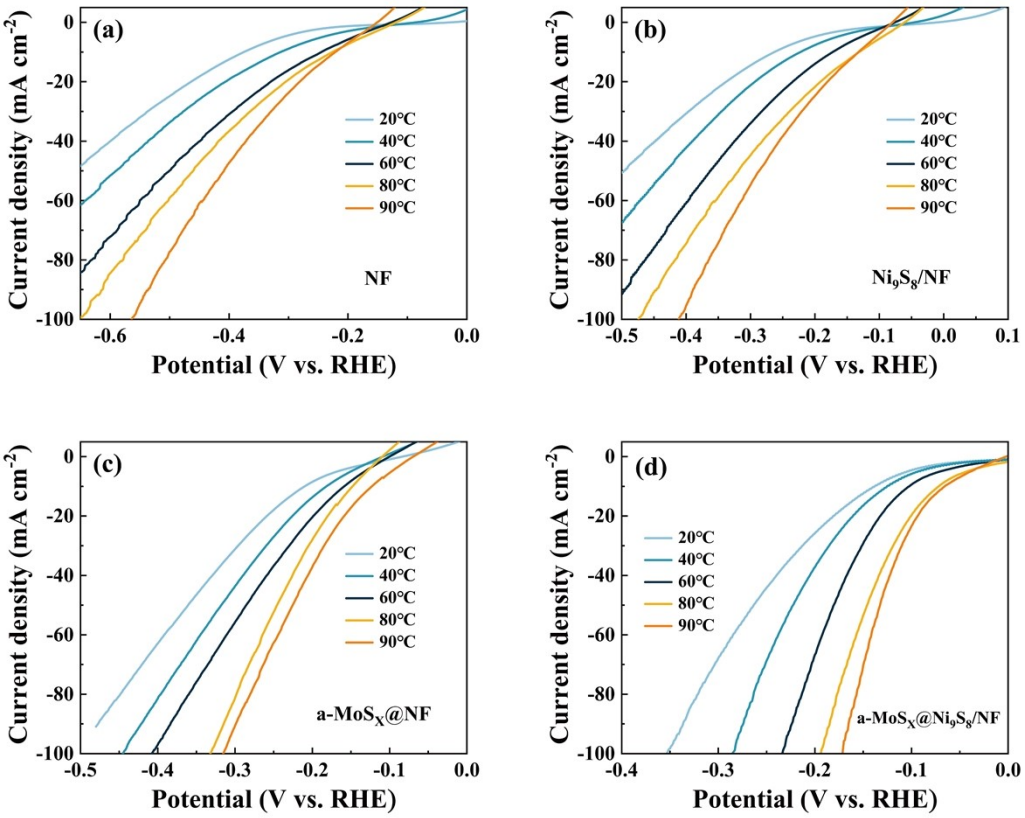

Fig. S3. LSV curves of (a) NF, (b) Ni_9S_8/NF, (c) a-MoS_X@NF and (d) a-MoS_X@Ni_9S_8/NF at 20, 40, 60, 80 and 90 °C, respectively.

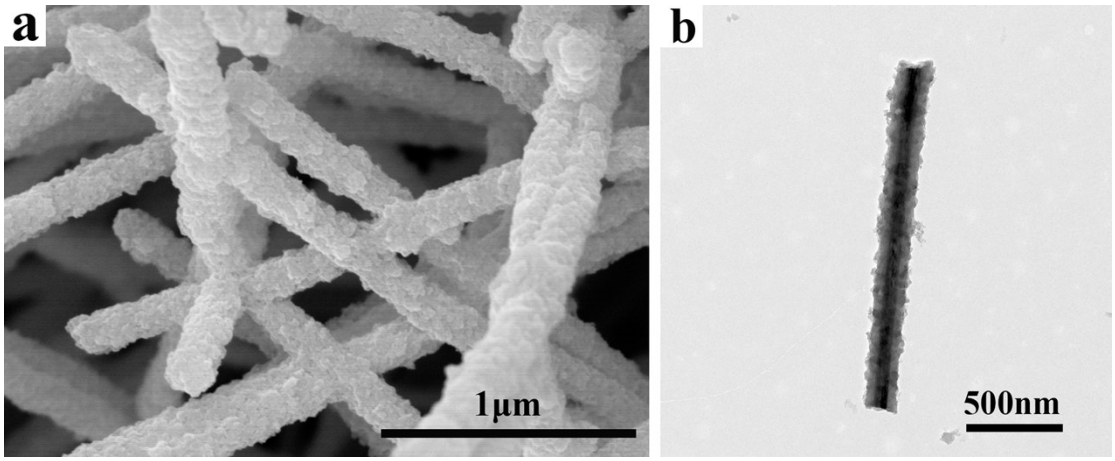

Fig. S4. (a) SEM and (b) TEM images of a-MoS_X@Ni_9S_8/NF after 5000 cycles.

Table S1. The BET surface area value of samples.

Sample	BET surface area/ $m^2 g^{-1}$
NF	0.051
Ni_9S_8/NF	4.610
a-MoS_X@Ni_9S_8/NF	4.773

Table S2. ECSA value of samples.

Sample	C_{dl}/ mF cm^{-2}	ECSA/ cm^2
NF	0.6	15.0
Ni_9S_8/NF	10.2	255.0
a-MoS_X@NF	20.9	522.5
a-MoS_X@Ni_9S_8/NF	50.8	1270.0

Table S3. R_{ct} value of samples.

Sample	R_{ct} / Ω
NF	417.07
Ni_9S_8/NF	8.48
a-MoS_X@NF	5.58
a-MoS_X@Ni_9S_8/NF	1.29