Supporting Information

Two-step Synthesis of Binary Ni-Fe Sulfides Supported on Nickel Foam as Highly Efficient Electrocatalysts for Oxygen Evolution Reaction

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Fig. S1 (a, b, c) HR-TEM images of NiFe/NF; (d) Selected area electron diffraction image of NiFe/NF.
**Fig. S2** (a, b, c) HR-TEM images of NiFeS-Fe/NF; (d) Selected area electron diffraction image of NiFeS-Fe/NF.
Fig. S3 CVs of the double-layer capacitance measurement for the four different samples in 1 M KOH in the non-Faradaic region of -0.05-0.05 V vs. SCE with different scan rates, varying from 40 mV s$^{-1}$ to 200 mV s$^{-1}$: (a) NiFe/NF; (b) NiFeS/NF; (c) NiFeS-Fe/NF and (d) Ni$_x$S$_y$/NF.
Calculation of electrochemical surface area (ECSA) and roughness factor (RF)\(^1,2\)

According to the previous literature, the electrochemical surface area (ECSA) and the roughness factor (RF) can be calculated based on following equations.

\[
\text{ECSA} = \frac{C_{dl}}{C_s}
\]

\[
\text{RF} = \frac{\text{ECSA}}{\text{GSA}}
\]

\(C_{dl}\): the measured double layer capacitance of NiFe/NF, NiFeS/NF and NiFeS-Fe/NF electrode in 1 M KOH (in mF);

\(C_s\): the specific capacitance, according to the literature, the value is 0.040 mF cm\(^{-2}\) in 1M NaOH;

\(\text{GSA}\): the geometric surface area of NiFe/NF, NiFeS/NF and NiFeS-Fe/NF electrode, which is 1 cm\(^2\)
Fig. S4 Stability test of NiFe/NF after 500 cycles.
**Fig. S5** (a, c, e) SEM image of NiFe/NF before stability test; (b, d, f) SEM image of NiFe/NF after stability test.
**Fig. S6** Stability test of NiFeS/NF and NiFeS-Fe/NF after 1000 cycles.
Fig. S7 (a) SEM image of NiFeS/NF before stability test of 1000 cycles; (b) SEM image of NiFeS/NF after stability test of 1000 cycles.
Fig. S8 (a, b) SEM image of NiFeS-Fe/NF before stability test of 1000 cycles; (c, d) SEM image of NiFeS-Fe/NF after stability test of 1000 cycles.
Table S1. The calculated ECSA and RF of the as-prepared NiFe/NF, Ni$_x$S$_y$/NF, NiFeS/NF and NiFeS-Fe/NF samples $^{1,2}$.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$C_{dl}$ (mF)</th>
<th>$C_s$ (mF cm$^{-2}$)</th>
<th>ECSA (cm$^2$)</th>
<th>GSA (cm$^2$)</th>
<th>RF</th>
</tr>
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<tbody>
<tr>
<td>NiFe/NF</td>
<td>6.95</td>
<td>0.04</td>
<td>173.75</td>
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<td>Ni$_x$S$_y$/NF</td>
<td>9.37</td>
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<td>NiFeS/NF</td>
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<td>0.04</td>
<td>251.25</td>
<td>1</td>
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<tr>
<td>NiFeS-Fe/NF</td>
<td>8.20</td>
<td>0.04</td>
<td>205.00</td>
<td>1</td>
<td>205.00</td>
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</tbody>
</table>
Reference
