Supporting Information

Hierarchically Nanostructured Nickel–cobalt Alloy Supported on Nickel Foam as a Highly Efficient Electrocatalyst for Hydrazine Oxidation

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Number of figures: 12
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**Figure S1.** (a,b) FE–SEM images at different magnifications of the hydrothermal sample.

**Figure S2.** (a–c) FE–SEM images at different magnifications of the one–step calcined sample at 300 °C under H₂ atmosphere for 1 h.
**Figure S3.** (a) LSV curves of Ni–Co/NF electrocatalysts prepared from the precursor mixtures with different Ni/Co molar ratios. The electrochemical tests were conducted in a solution containing 0.5 M N\textsubscript{2}H\textsubscript{4}·H\textsubscript{2}O and 1.0 M NaOH at a scan rate of 20 mV s\textsuperscript{-1}. The inset shows a zoomed–in view of the onset potential region of the examined electrocatalysts. (b) Current density and onset potential of Ni–Co/NF electrocatalysts derived from figure a.
Figure S4. (a) LSV curves of Ni–Co/NF electrocatalysts that were annealed at different temperatures under reductive atmosphere. The electrochemical tests were conducted in a solution containing 0.5 M N₂H₄·H₂O and 1.0 M NaOH at a scan rate of 20 mV s⁻¹. The inset shows a zoomed–in view of the onset potential region of the examined electrocatalysts. (b) Current density and onset potential of Ni–Co/NF electrocatalysts derived from figure a.
Figure S5. FE–SEM images at different magnifications of (a–c) Ni/NF and (d–f) Co/NF catalysts.

Figure S6. (a–c) FE–SEM images at different magnifications of the Ni–Co/NF catalyst after 1000 CV cycles in a solution containing 3.0 M N$_2$H$_4$·H$_2$O and 1.0 M NaOH.
Figure S7. (a) Ni 2p and (b) Co 2p spectrum of the Ni–Co/NF sample before and after 1000 electrochemical cycling in a solution containing 3.0 M N₂H₄·H₂O and 1.0 M NaOH.
**Figure S8.** XRD pattern of the Ni–Co/NF catalyst after 1000 CV cycles in a solution containing 3.0 M N₂H₄·H₂O and 1.0 M NaOH.
Figure S9. A comparison of the LSV curves of the Ni−Co/NF catalyst in 1 M NaOH electrolyte with and without \( \text{N}_2\text{H}_4\cdot\text{H}_2\text{O} \) at a scan rate of 20 mV·s\(^{-1}\).

It was observed that the Ni−Co/NF catalyst showed no significant anodic current in 1.0 M NaOH solution at the applied potential condition, indicating that the high anodic current in 1.0 M NaOH + 0.5 M \( \text{N}_2\text{H}_4\cdot\text{H}_2\text{O} \) solution could be safely attributed to the hydrazine electrooxidation reaction.
Figure S10. LSV curves of the Ni−Co/NF catalyst before and after a long−term operation under 10mA cm−2. The electrochemical tests were conducted in a solution containing 0.5 M N₂H₄·H₂O and 1.0 M NaOH at a scan rate of 20 mV s⁻¹.
Figure S11. XRD patterns of the powdery samples that were peeled off from the Ni foam substrate of the Co/NF and Ni/NF catalysts.
Figure S12. Comparison of the LSV curves of Ni–Co/NF and the "state of the art" Ni$_2$P@Ni$_{10}$Mo/Ni–Mo–O/NF catalyst [13]. The electrochemical tests were conducted in a solution containing 0.5 M N$_2$H$_4$·H$_2$O and 1.0 M NaOH at a scan rate of 20 mV s$^{-1}$. 
Table S1. A comparison of catalytic activities of various catalysts towards hydrazine electrooxidation.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Electrolyte [N$_2$H$_4$] and [NaOH] (M)</th>
<th>Electrocatalytic performance</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni−NSA/NF</td>
<td>1.0, 3.0</td>
<td>228</td>
<td>0.25</td>
</tr>
<tr>
<td>Cu–Ni/Cu foil</td>
<td>0.1, 3.0</td>
<td>300</td>
<td>0.45</td>
</tr>
<tr>
<td>Ni$<em>{0.6}$Co$</em>{0.4}$−ANS A/NF</td>
<td>0.5, 3.0</td>
<td>250</td>
<td>0.20</td>
</tr>
<tr>
<td>Ni−Zn/NF</td>
<td>0.1, 1.0</td>
<td>300</td>
<td>0.40</td>
</tr>
<tr>
<td>Ni−Zn/NF</td>
<td>0.1, 1.0</td>
<td>370</td>
<td>0.30</td>
</tr>
<tr>
<td>Pd−porous Ni/NF</td>
<td>0.1, 1.0</td>
<td>450</td>
<td>0.30</td>
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<tr>
<td>Ni$_2$P/NF</td>
<td>0.1, 1.0</td>
<td>220</td>
<td>0.10</td>
</tr>
<tr>
<td>Ni$_3$P/NF (DP)</td>
<td>0.1, 1.0</td>
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<td>0.30</td>
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<td>NiS$_2$/TiM</td>
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<td>300</td>
<td>0.22</td>
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<tr>
<td>CoNi−S/NF</td>
<td>2.0, 0.1</td>
<td>118</td>
<td>1.0</td>
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<td>Ni−B/NF</td>
<td>0.1, 1.0</td>
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<td>0.30</td>
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<td>Ni$_3$N/Ni/NF</td>
<td>0.5, 1.0</td>
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<td>0.30</td>
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<td>Ni$<em>3$P@Ni$</em>{10}$Mo/Ni−Mo−O/NF</td>
<td>0.5, 1.0</td>
<td>822</td>
<td>0.30</td>
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<td>Ni−Co/NF</td>
<td>0.5, 1.0</td>
<td>1213</td>
<td>0.30</td>
</tr>
</tbody>
</table>

This work
References


