

**Supplementary Information for**

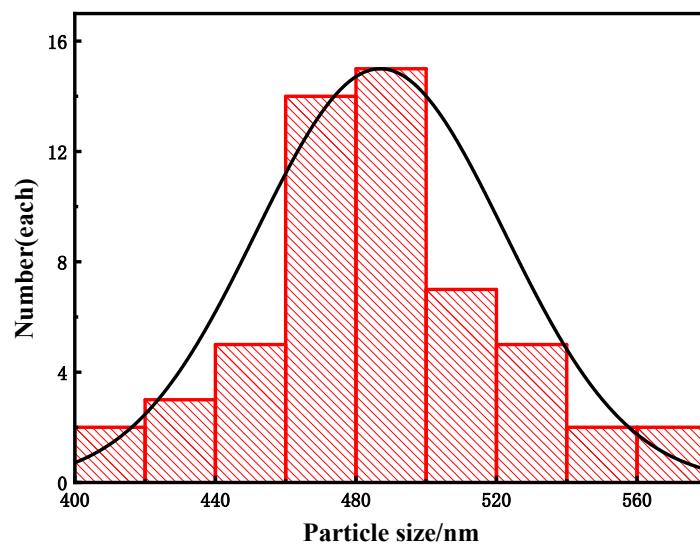
**Molybdenum in-situ etching treated ultra-thin NiFeMo LDHs nanosheet arrays as performance anodic catalyst for efficient industrial hydrogen production**

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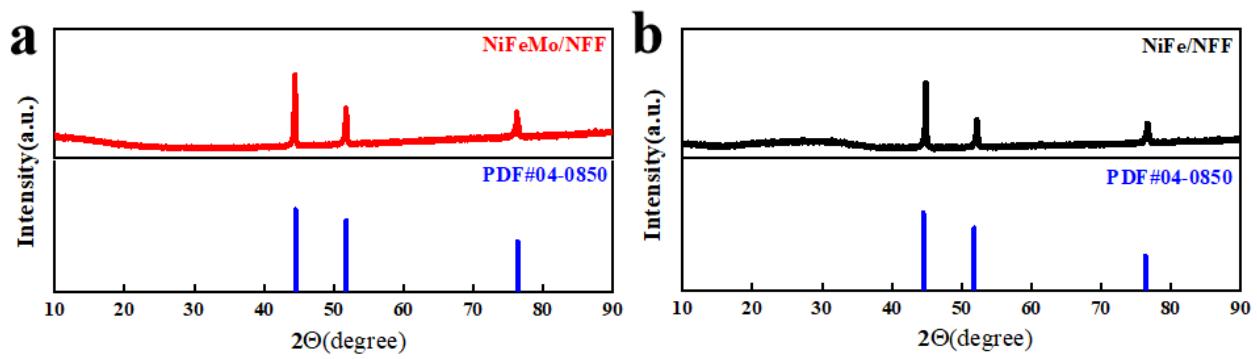
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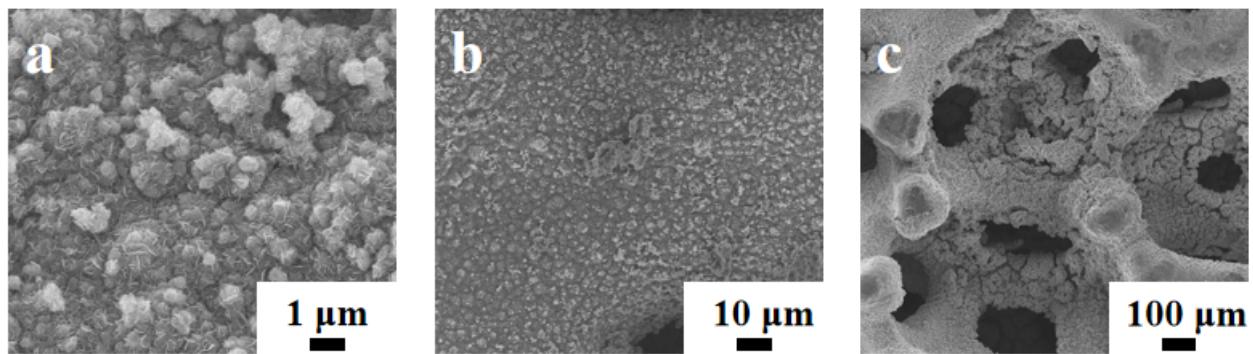
**Figure S1** Normal distribution histogram of particle size for NiFeMo/NFF



**Figure S2** XRD profile of NiFeMo/NFF(a) and NiFe/NFF(b)

**Figure S3** LSV curve and overpotential comparison of NFF, NiFe/NFF, NiFeMo/NFF

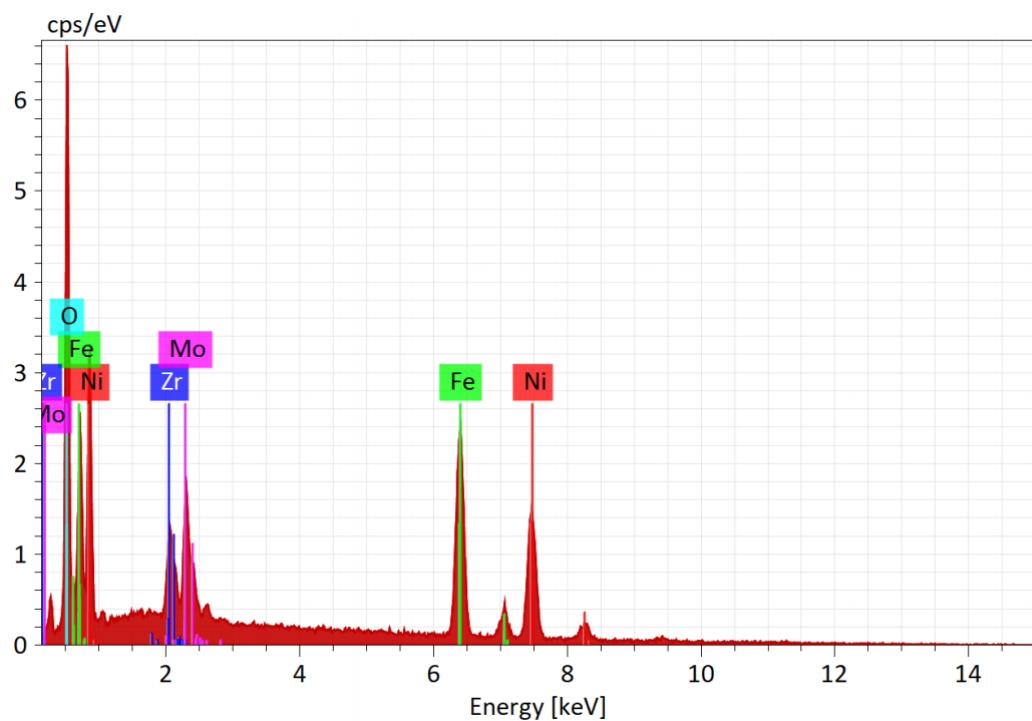
**Figure S4** The CV curves of NFF, NiFe/NFF, NiFeMo/NFF



**Figure S5** SEM characterization of NiFeMo/NFF after long-term electrolysis

**Table S1** EDS Mapping of NiFeMo/NF

| Element | Atomic Number | Mass (%) | Atomic (%) |
|---------|---------------|----------|------------|
| O       | 8             | 22.99    | 53.32      |
| Fe      | 26            | 29.69    | 22.06      |
| Ni      | 28            | 34.91    | 19.72      |
| Mo      | 42            | 7.74     | 2.99       |
| Zr      | 40            | 4.68     | 1.90       |



**Table S2** Overpotential Data of Different Materials at 10 to 1000 mA cm<sup>-2</sup>

| <b>Electrode</b>  | <b>10 mA cm<sup>-2</sup></b> | <b>100 mA cm<sup>-2</sup></b> | <b>300 mA cm<sup>-2</sup></b> | <b>500 mA cm<sup>-2</sup></b> | <b>800 mA cm<sup>-2</sup></b> | <b>1000 mA cm<sup>-2</sup></b> |
|-------------------|------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|--------------------------------|
| <b>NiFeMo/NFF</b> | <b>288 mV</b>                | <b>367 mV</b>                 | <b>476 mV</b>                 | <b>575 mV</b>                 | <b>712 mV</b>                 | <b>784 mV</b>                  |
| <b>NiFe/NFF</b>   | <b>296 mV</b>                | <b>392 mV</b>                 | <b>518 mV</b>                 | <b>627 mV</b>                 | <b>779 mV</b>                 | <b>848 mV</b>                  |
| <b>NFF</b>        | <b>327 mV</b>                | <b>423 mV</b>                 | <b>564 mV</b>                 | <b>689 mV</b>                 | <b>866 mV</b>                 | <b>975 mV</b>                  |

**Table S3** Cdl and ECSA

| <b>Electrode</b>  | <b>Cdl (mF cm<sup>-2</sup>)</b> | <b>ECSA (cm<sup>2</sup>)</b> |
|-------------------|---------------------------------|------------------------------|
| <b>NiFeMo/NFF</b> | <b>14.28</b>                    | <b>357</b>                   |
| <b>NiFe/NFF</b>   | <b>3.36</b>                     | <b>84</b>                    |
| <b>NFF</b>        | <b>0.94</b>                     | <b>23.5</b>                  |

**Table S4** AC Impedance

| <b>Electrode</b>  | <b>Rs</b>   | <b>Rp</b>    |
|-------------------|-------------|--------------|
| <b>NiFeMo/NFF</b> | <b>0.70</b> | <b>6.88</b>  |
| <b>NiFe/NFF</b>   | <b>0.72</b> | <b>36.56</b> |
| <b>NFF</b>        | <b>0.70</b> | <b>219.7</b> |

**Table S5** Electrochemical properties of Ni/Fe based catalysts in recent years

| catalytic agent  | Synthesis method                            | Electrochemical tests                     |                        |  | Ref       |
|--|---|---|------------------------|--|-----------|
|  |   | Current Density<br>(mA cm <sup>-2</sup> ) | Over potential<br>(mV) | Tafel slope<br>(mV dec <sup>-1</sup> ) |           |
| NiFe-LDH/Ti <sub>3</sub> C <sub>2</sub>                | hydro-thermal method                        | 10  | 334                    | 55                                     | 1         |
| NiFe-LDH/NaMnO   | molten-salt growth method                   | 20  | 260                    | 21                                     | 2         |
| Mo–NiFe <sub>2</sub> O <sub>4</sub> –V <sub>O</sub>    | High temperature calcination method         | 10  | 315                    | 35.9                                   | 3         |
| Fe <sub>0.3</sub> Ni <sub>1</sub> Co <sub>2</sub> /S-C | hydro-thermal method<br>Radio frequency     | 10  | 276                    | 52.2                                   | 4         |
| NiFeCo oxide   | magnetron sputtering technology             | 10  | 280                    | 32.25                                  | 5         |
| FeCoMo/CP  | Co-precipitation + pyrolysis<br>Single-step | 10  | 270                    | 63                                     | 6         |
| FeNiVO <sub>x</sub>                                    | aerosol assisted chemical vapor deposition  | 10  | 250                    | 51                                     | 7         |
| FeCoMn/PC  | Solvent thermal method                      | 10  | 170                    | 66.7                                   | 8         |
| NiFeMo/NFF   | Electrochemical in situ etching method      | 10  | 288                    | 43.5                                   | This work |

## **References:**

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