Supporting Information for

Ultrathin Nickel-Iron Layered Double Hydroxide Nanosheets Intercalated with Molybdate Anions for Electrocatalytic Water Oxidation

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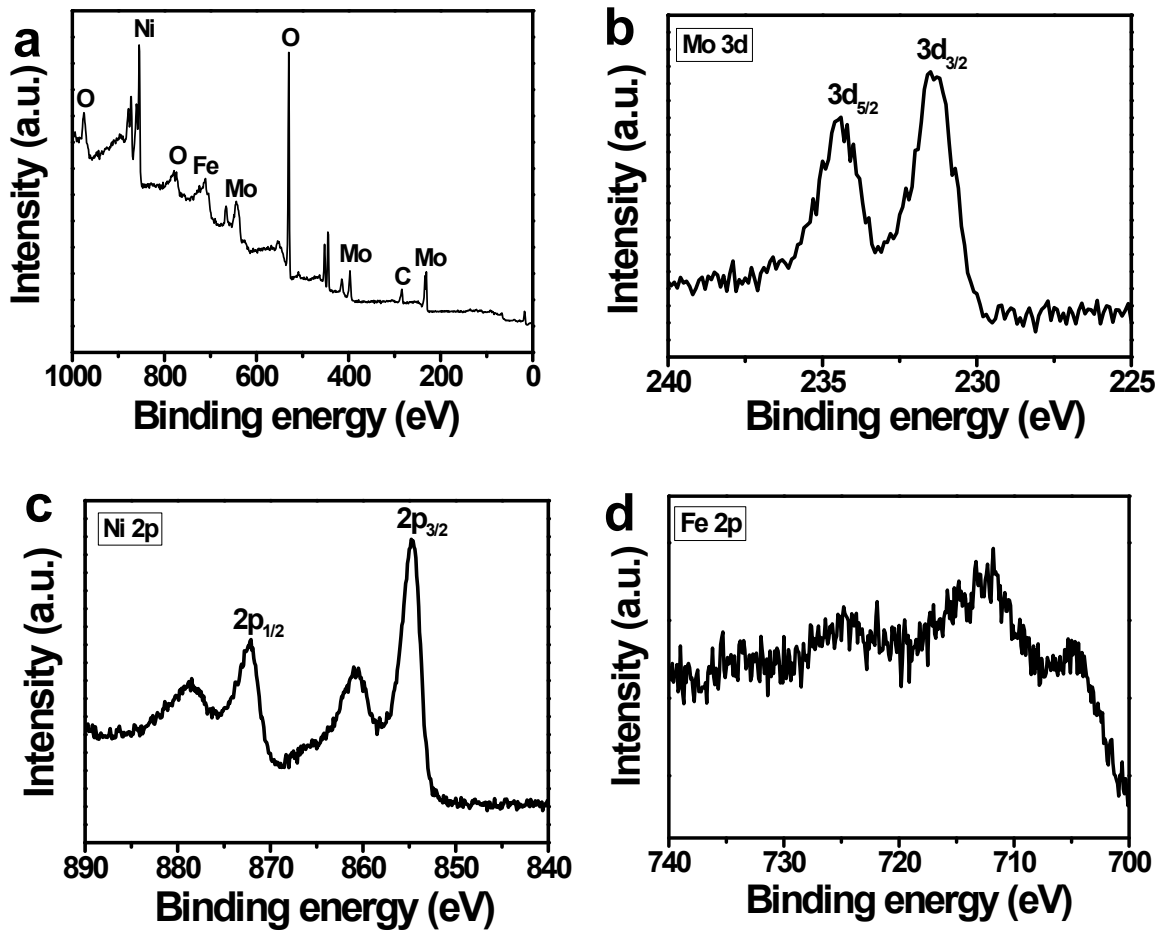

Figure S1. (a) XPS survey spectrum, (b) high-resolution Mo 3d spectrum, (c) Ni 2p spectrum and (d) Fe 2p spectrum of NiFeMo nanosheets.

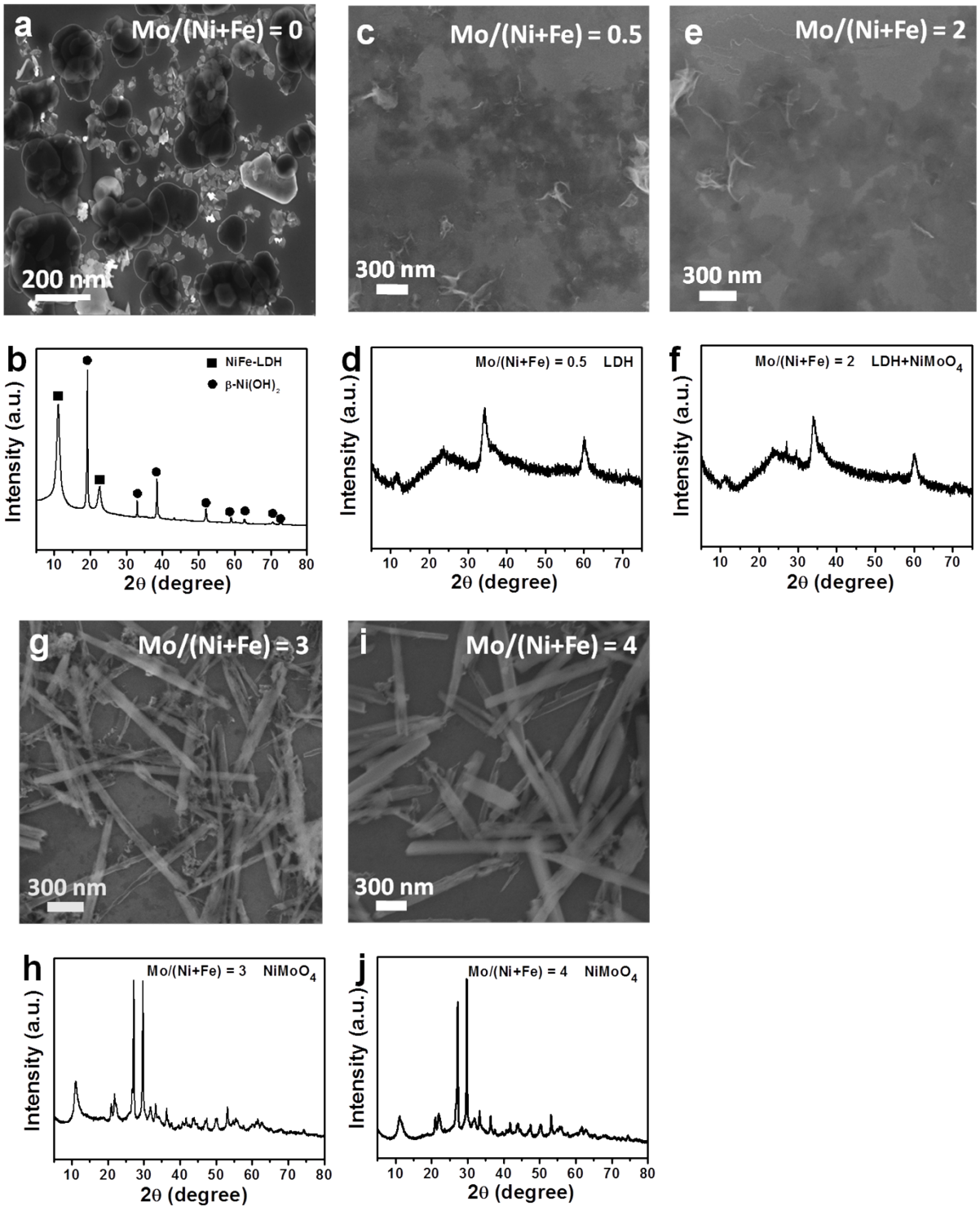
a
Mo/(Ni+Fe) = 0
200 nm
c
Mo/(Ni+Fe) = 0.5
300 nm
e
Mo/(Ni+Fe) = 2
300 nm
b
NiFe-LDH
β-Ni(OH)2
Intensity (a.u.)
2θ (degree)
d
Mo/(Ni+Fe) = 0.5 LDH
Intensity (a.u.)
2θ (degree)
f
Mo/(Ni+Fe) = 2 LDH+NiMoO4
Intensity (a.u.)
2θ (degree)
g
Mo/(Ni+Fe) = 3
300 nm
i
Mo/(Ni+Fe) = 4
300 nm
h
Mo/(Ni+Fe) = 3 NiMoO4
Intensity (a.u.)
2θ (degree)
j
Mo/(Ni+Fe) = 4 NiMoO4
Intensity (a.u.)
2θ (degree)

Figure S2. The effect of different starting Mo/(Ni+Fe) molar ratios on the morphology and crystal structure of final products.

Table S1. A brief survey of recent works on NiFe-LDH OER electrocatalysts.

Reference	**OER electrocatalysts**	**Loading (mg/cm^2)**	**Overpotential at j = 10mA/cm^2 (mV)**	**Tafel slope (mV/dec)**
This study	Ultrathin NiFe LDH nanosheets with MoO_4^{2-} intercalation	0. 28	280	40
Nat. Commun. **2014**, *5*, 4477.	LDH monolayers from solution exfoliation	0.07	300	58
J. Am. Chem. Soc. **2014**, *136, 6744*.	Electrodeposited Ni-Fe hydroxide films	N/A	300 mV for j = 5 mA/cm^2	N/A
J. Am. Chem. Soc. **2013**, *135, 12329*.	Electrodeposited Ni-Fe hydroxide films on gold electrodes	N/A	300	40
J. Am. Chem. Soc. **2013**, *135, 8452*.	NiFe-LDH nanoplates	0.2	320	N/A
	NiFe-LDH nanoplates grown on MWCNTs	0.2	230	31

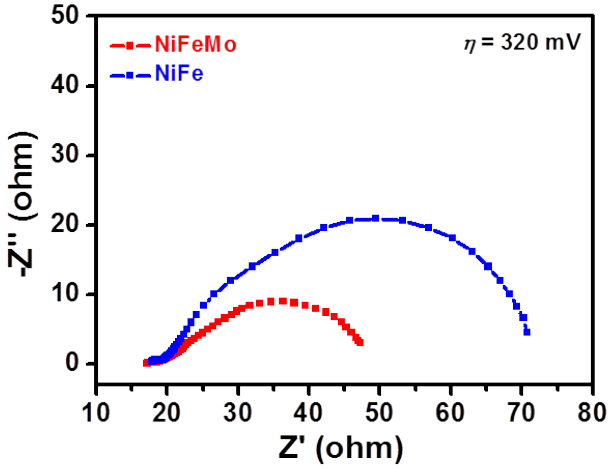

Figure S3. Electrochemical impedance spectra of NiFeMo and NiFe for OER electrocatalysis at η = 320 mV.

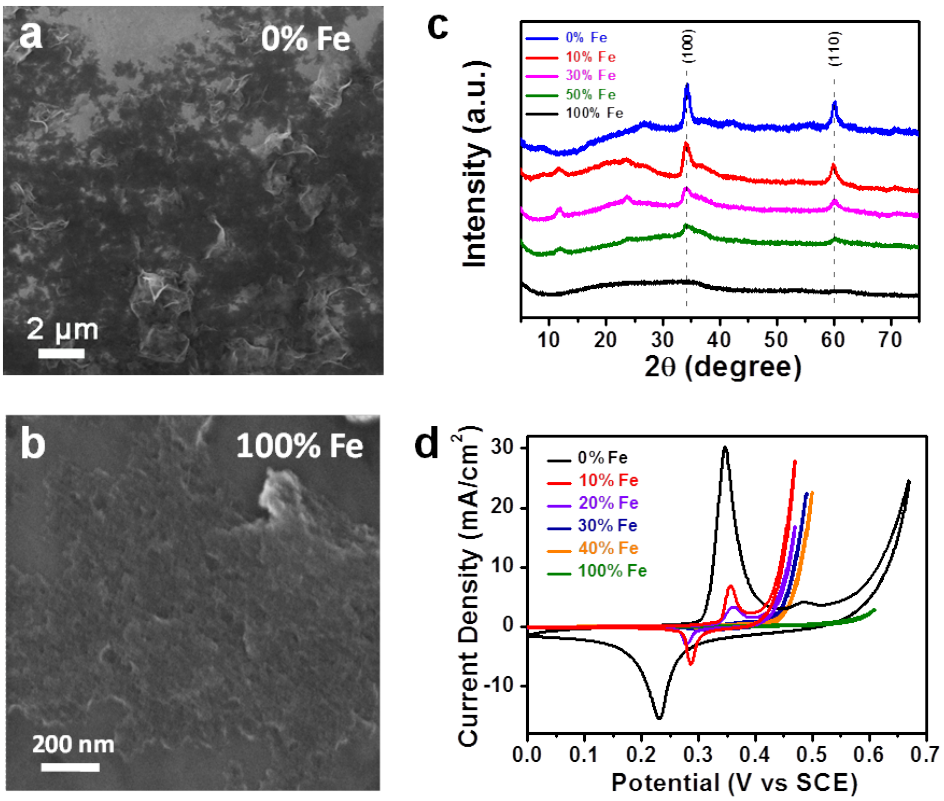

Figure S4. The effect of different Fe at% on the morphology, crystal structure and electrochemical

performance of the final products. (a-b) SEM images of the final products with (a) 0% Fe and (b) 100% Fe. (c) XRD and (d) CV curves of the final products with different Fe contents. Mo/(Ni+Fe) is fixed at 0.1.