Joule heating-assisted preparation of high-efficiency, long-term stability FeNiMoO hydrogen evolution reaction catalyst

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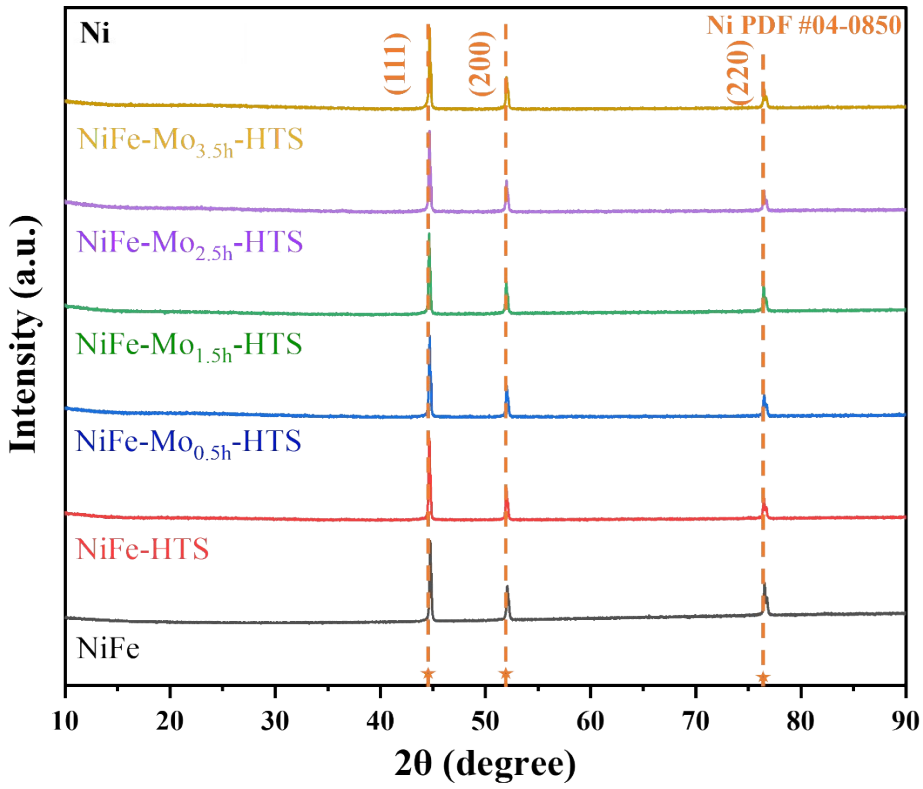

Figure S1. XRD images of NiFe, NiFe-HTS, and NiFeMo-HTS samples under different ammonium molybdate etching times.

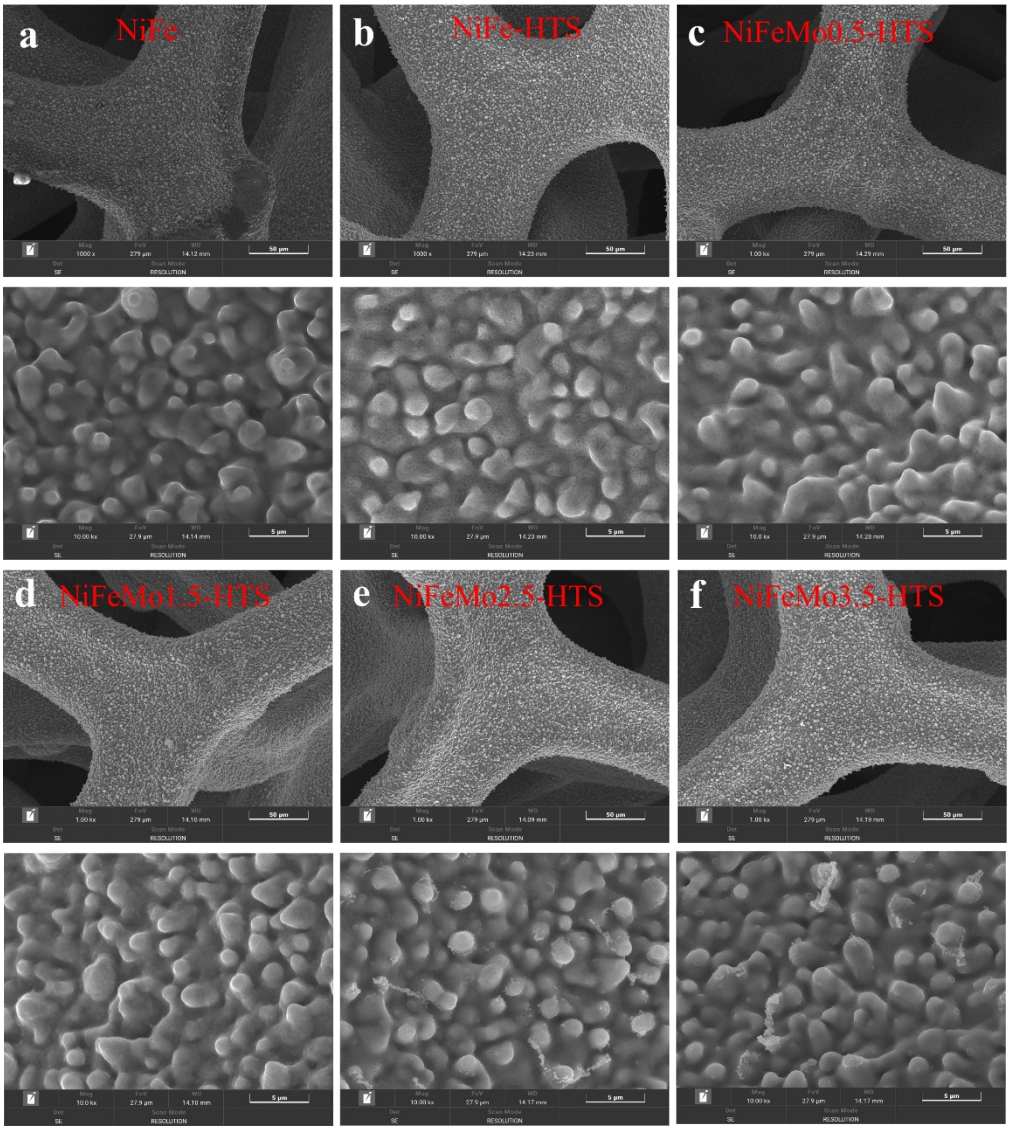

Figure S2. SEM images of the catalysts. (a) NiFe, (b) NiFe-HTS, (c) NiFe-$Mo_{0.5h}$-HTS, (d) NiFe-$Mo_{1.5h}$-HTS, (e) NiFe-$Mo_{2.5h}$-HTS, (f) NiFe-$Mo_{3.5h}$-HTS.

Table S1. The atomic percentage of the NiFe-Mo_{xh}-HTS（x=0, 0.5, 1.5, 2.5, 3.5）

	O	Mo	Fe	Ni	Molar ratio
NiFe	1.12	/	1.75	97.13	≈ 0:0:1:56
NiFe-HTS	13.25	/	1.54	85.21	≈ 9:0:1:55
NiFe-$Mo_{0.5h}$-HTS	13.29	0.67	1.92	84.12	≈ 7:0:1:44

NiFe-$Mo_{1.5h}$-HTS	18.52	4.11	1.45	75.91	≈ 13:3:1:52
NiFe-$Mo_{2.5h}$-HTS	19.15	4.74	1.36	74.74	≈ 14:3:1:55
NiFe-$Mo_{3.5h}$-HTS	28.61	3.9	1.96	65.53	≈ 15:2:1:33

Table S2. The mass fraction of the NiFe-Mo_{xh}-HTS（x=0, 0.5, 1.5, 2.5, 3.5）

	O	Mo	Fe	Ni
NiFe	0.31	/	1.68	98.01
NiFe-HTS	4.00	/	1.63	94.37
NiFe-$Mo_{0.5h}$-HTS	4.00	1.20	2.02	92.79
NiFe-$Mo_{1.5h}$-HTS	5.67	7.54	1.55	85.24
NiFe-$Mo_{2.5h}$-HTS	5.86	8.71	1.46	83.97
NiFe-$Mo_{3.5h}$-HTS	9.56	7.82	2.28	80.34

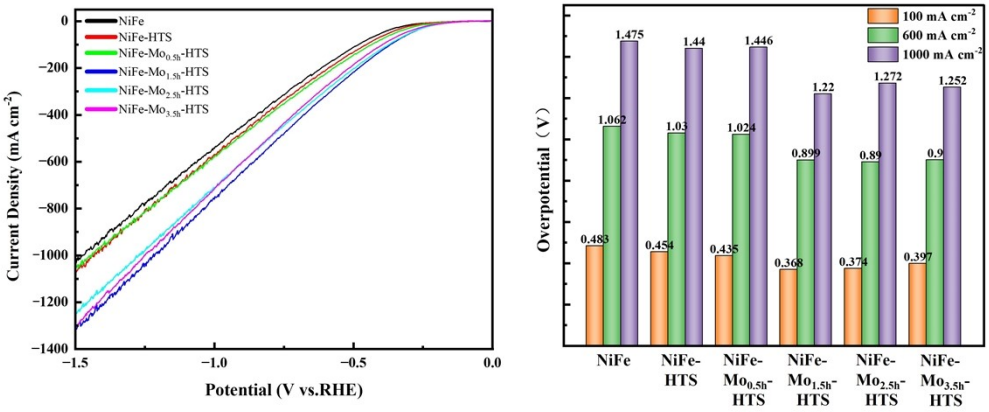

Figure S3. LSV curves of NiFe, NiFe-HTS, and NiFeMo-HTS samples under different

ammonium molybdate etching times.

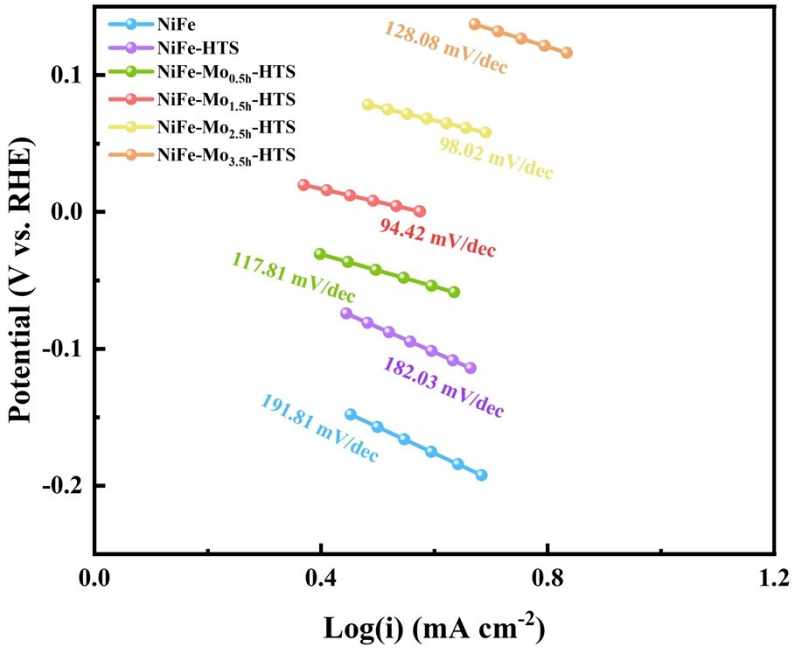

Figure S4. Tafel slopes of NiFe, NiFe-HTS, and NiFeMo-HTS samples under different ammonium molybdate etching times.

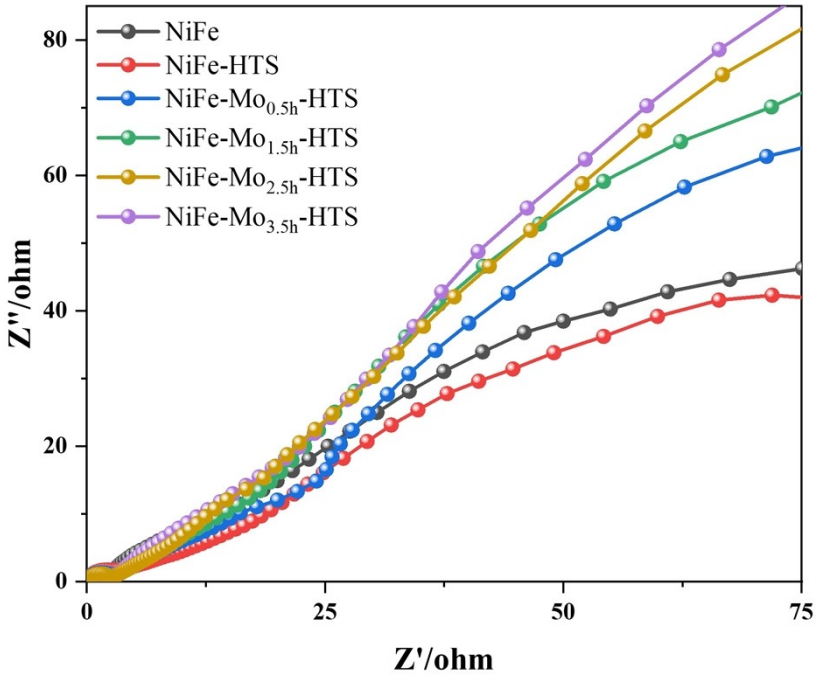

Figure S5. EIS of NiFe, NiFe-HTS, and NiFeMo-HTS samples under different ammonium molybdate etching times.

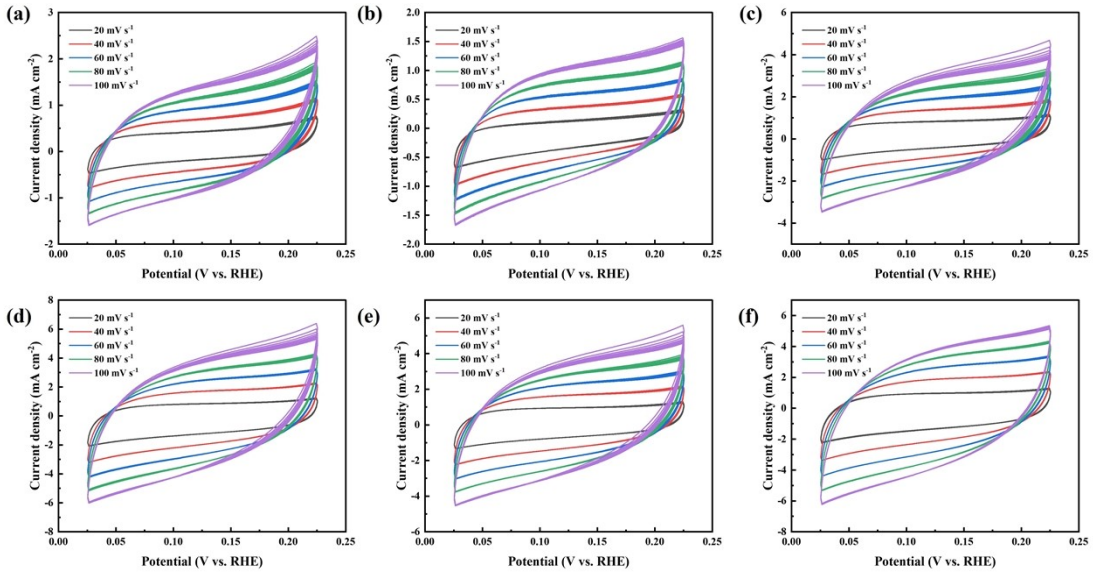

Figure S6. CV curves of (a) NiFe, (b) NiFe-HTS, (c) NiFe-$Mo_{0.5h}$-HTS, (d) NiFe-$Mo_{1.5h}$-HTS, (e) NiFe-$Mo_{2.5h}$-HTS and (f) NiFe-$Mo_{3.5h}$-HTS.

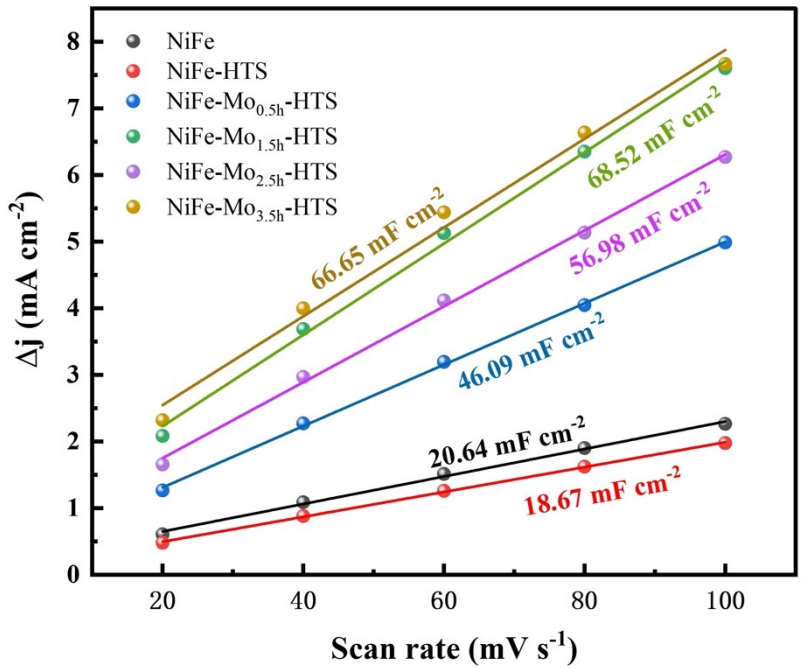

Figure S7. Cdl of NiFe, NiFe-HTS, and NiFeMo-HTS samples under different ammonium molybdate etching times.

Table S3. The comparison between this work and other catalysts for HER stability.

Catalysts	Potential	Stability	ECSA	iR correction	Preparation method	Reference
$NiFeMo_{1.5h}$-HTS	1000mA cm^{-2}, 1220mV	400mA cm^{-2}, 3000h	1713	No	Joule heating	This work
NiFeMo IOS	10mA cm^{-2}, 33mV	500mA cm^{-2}, 50h	/	No	Electro-deposition	1
$Ni_2P/CoP/FeP_4$ /IF	100mA cm^{-2}, 127mV	500mA cm^{-2}, 25h	812.15	No	Hydro-thermal	2
NiFe-LDH	10mA cm^{-2}, 88.5mV	500mA cm^{-2}, 12h	1.775	No	Hydro-thermal	3
FeCoP/NiFe LDH	100mA cm^{-2}, 153mV	100mA cm^{-2},100h	391.5	Yes	Hydro-thermal	4
Ni/MnO	10mA cm^{-2}, 114mV	500mA cm^{-2}, 72h	1113.5	No	Copre-cipitation	5
Ru-Co/NCW	1000mA cm^{-2}, 242mV	100mA cm^{-2}, 100h	64.7	Yes	High-temperature nitridation	6
CuO@NiO/Ni	100mA cm^{-2}, 86.5mV	500mA cm^{-2}, 100h	301.25	No	Electro-deposition	7
Ru_{SA}/NiFe-LDH/MoNiS	500mA cm^{-2}, 234mV	500mA cm^{-2}, 200h	579.25	No	High-temperature pyrolysis	8
NiRu@C	10mA cm^{-2}, 33mV	1000mA cm^{-2}, 200h	/	No	High-temperature pyrolysis	9
Ni_2V/V	100mA cm^{-2}, 200mV	500mA cm^{-2}, 100h	2105.3	No	High-temperature solid-state reaction	10
Pd-CoNiP/NF	100mA cm^{-2}, 65mV	100mA cm^{-2},60h	179.83	Yes	Heat treatment	11
PtNiCuCoPd	10mA cm^{-2}, 38mV	200mA cm^{-2},96h	1908	No	Magnetron sputtering	12

AlNbPt	100mA cm^{-2}, 69mV	800mA cm^{-2}, 100h	809	Yes	High-temperature melting / quenching	[13]
Vo-NiMoOOH/NF	/	500mA cm^{-2}, 150h	/	No	Hydro-thermal	[14]
Rh/Co-NDC	/	1000mA cm^{-2}, 550h	55	No	Solvo-thermal synthesis	[15]

Table S4. The atomic percentage of the NiFe-$Mo_{1.5h}$-HTS after stability test

	O	Mo	Fe	Ni	Molar ratio
Before	18.52	4.11	1.45	75.91	≈ 13:3:1:52
After	7.33	5	2.16	85.48	≈ 3:2:1:40

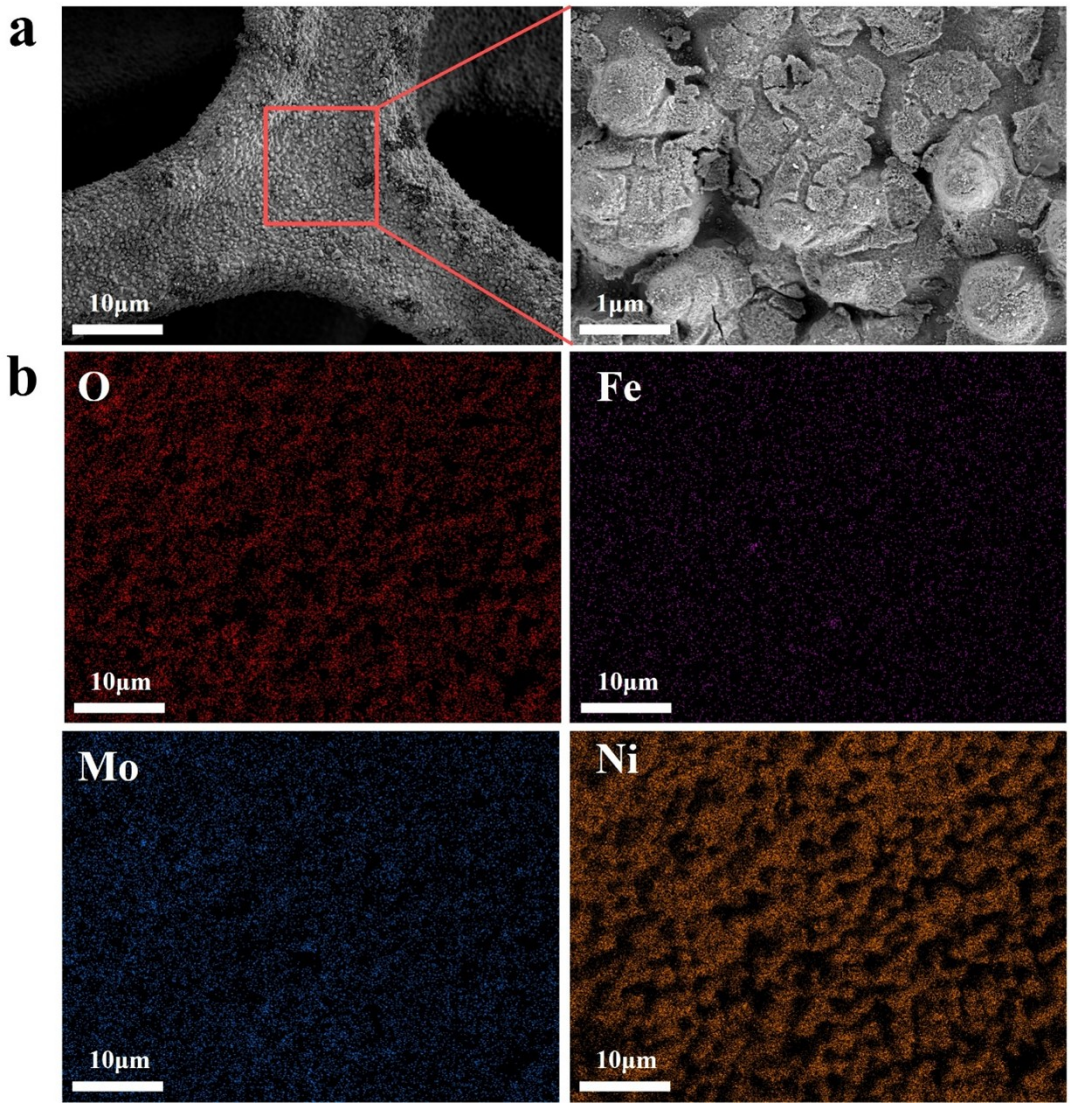

Figure S8. SEM images (a) and corresponding EDS mappings (b) of NiFe-$Mo_{1.5h}$-HTS after the stability test.

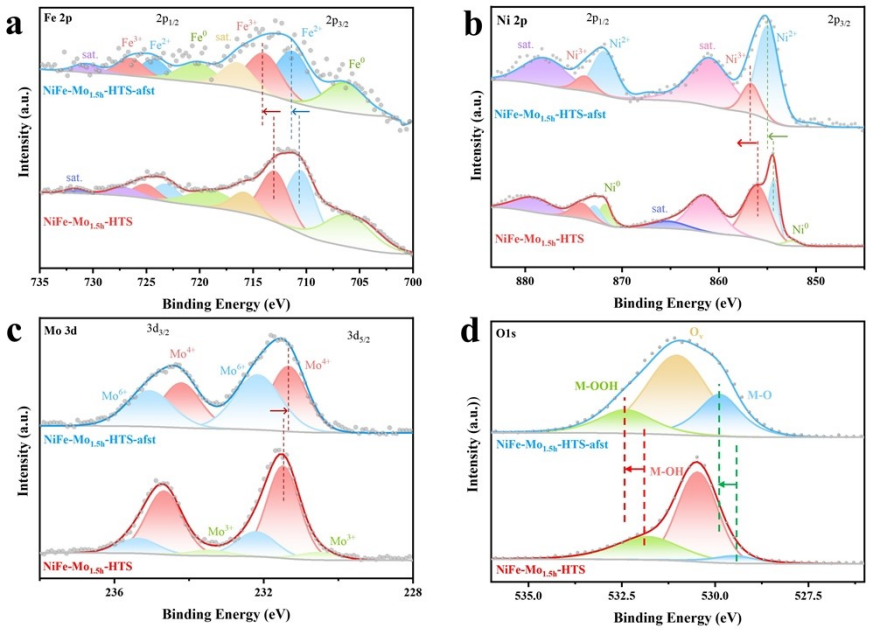

Figure S9. High -resolution XPS spectra of (a) Fe 2p, (b) Ni 2p, (c) Mo 3d and (d) O 1s before and after stability test.

Comment [PZ]: 稳定性后 Ni0 的不要了

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