Dual-strategy engineered nickel phosphide for achieving efficient hydrazine-assisted hydrogen production in seawater

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Experimental

Reagents and Materials. $Ni(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_2 \cdot 6H_2O$, NaH_2PO_4, Na_2HPO_4, NH_4F, $Ce(NO_3)_2 \cdot 6H_2O$, NaH_2PO_2, HCl, hydrazine, ethanol and urea were obtained from Sinopharm Chemical Reagent Co. Ltd. KOH was purchased from Adamas-beta®.

Synthesis of Fe-Ni$_2$P/CeO$_2$. Typically, $Ni(NO_3)_2$ (1.7 mmol), $Fe(NO_3)_2$ (0.3 mmol), $Ce(NO_3)_2$ (0.2 mmol), urea (10 mmol) and NH_4F (6 mmol) were dissolved in 35 mL water to gain homogeneous solution. Then, a piece of pretreated NF was immersed into above solution, and reacted at 120 ℃ to obtain NiFeCe layer double hydroxide (NiFeCe LDH) precursor. After that, the Fe-Ni$_2$P/CeO$_2$ product can be yielded by the phosphorization process at 300 °C with NiFeCe LDH and NaH_2PO_2 as precursor and phosphorus source. The control Ni$_2$P and Fe-Ni$_2$P samples were prepared in a similar procedure in the absence of $Fe(NO_3)_2$, $Ce(NO_3)_2$ and $Ce(NO_3)_2$, respectively.

Materials characterization. The X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray photoelectron spectrometer (XPS) were performed on the Bruker D8, FIB-SEM GX4, FEI Tecnai G20 instruments and Thermo ESCALAB 250 to research detailed phases, morphology, chemical composition and valences of synthesized samples.

Electrochemical measurements. Electrochemical behaviors were assessed on an electrochemical station (CHI 760E) employing a typical three-electrode system with graphite rod, saturated calomel electrode and synthesized samples as counter, reference and working electrodes in 1 M KOH seawater without and with 0.1-0.7 M N_2H_4 and 1 M PBS buffered seawater solution. For electrocatalytic tests in seawater, natural seawater was acquired from the Huanghai Sea, which was filtered to remove some impurities. The polarization curves were conducted at a scan rate of 2 mV s_{-1} and the corresponding potentials were calibrated to standard potentials *via* the nernst equation. The electrochemical impedance spectroscopy was measured in the frequency varying from 0.01 Hz to 100 kHz, respectively.

Theoretical calculations. The density functional theory calculations were carried out employing the Vienna Ab-initio Simulation Package (VASP) software.[1,2] The generalized gradient approximation by Perdew-Burke-Ernzerhor was applied to exchange-correlation energy.[3,4] The Kohn-Sham one-electron states were expanded using plane wave basis set with kinetic cutoff energy (500 eV). The active Fe-Ni$_2$P/CeO$_2$ surface was simulated by a periodic six-layer slab repeated in 2 ×1 surface unit cell. The Brillouin-zone integration was carried out by Gamma sampling method. A vacuum layer of 15 Å was adopted to avoid the interaction between neighboring slabs. To acquire Fe-Ni$_2$P/CeO$_2$ heterostructure interface, the CeO$_2$ (111) surface was modeled by a periodic three-layer slab with the O-termination. The origin Fe-Ni$_2$P (001) slab was expanded by a 2 ×2 supercell. After that, the Fe-Co$_2$P/CeO$_2$ interface was created by placing Fe-Ni$_2$P (001) slab onto CeO$_2$ (111) slab. To simplify the calculation, the interface cell with (010) surface was cleaved. The Brillouin-zone (BZ) integration was carried out using Gamma sampling method with a density of 3 × 2 × 1 for geometry optimization.

Adsorption energy was calculated as following:

$G=E + E_{ZPE} - TS$

Where the E, E_{ZPE} and S denote the adsorption energy, the difference of zero-point energy and the entropy change, which can be get from the vibration energy of intermediates and gas molecules.[5]

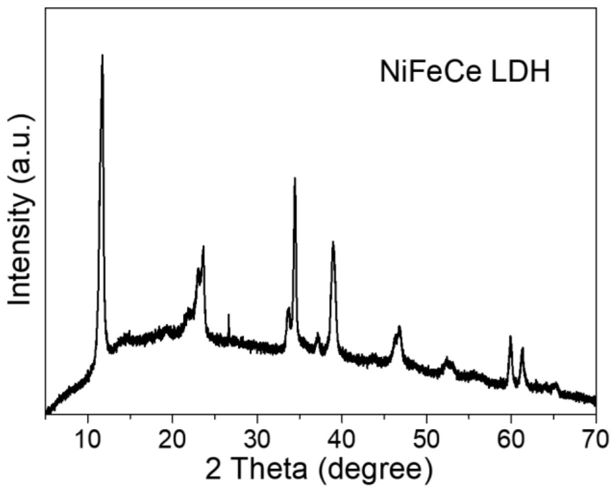

Fig. S1 XRD pattern of NiFeCe LDH precursor.

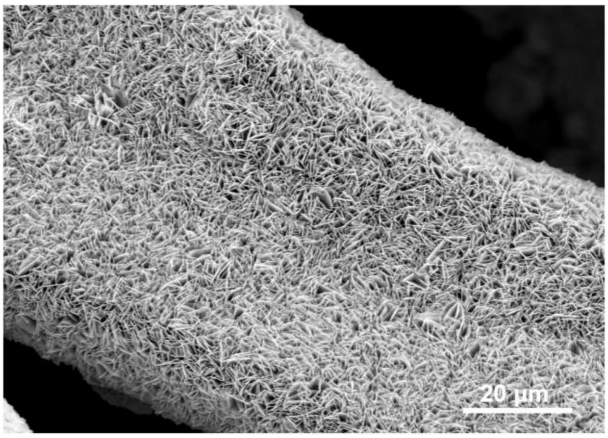

Fig. S2 SEM image of Fe-Ni_2P/CeO_2.

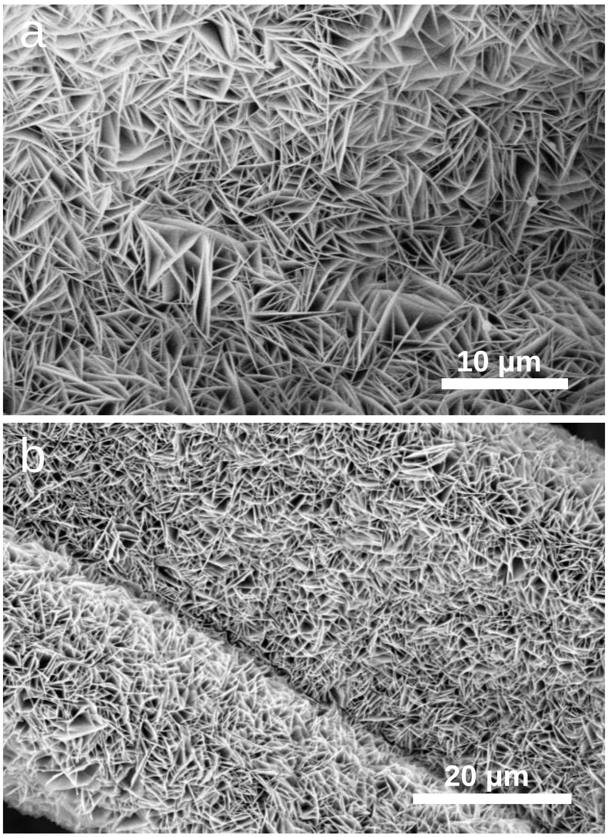

Fig. S3 SEM images of (a) Ni_2P and (b) Fe-Ni_2P.

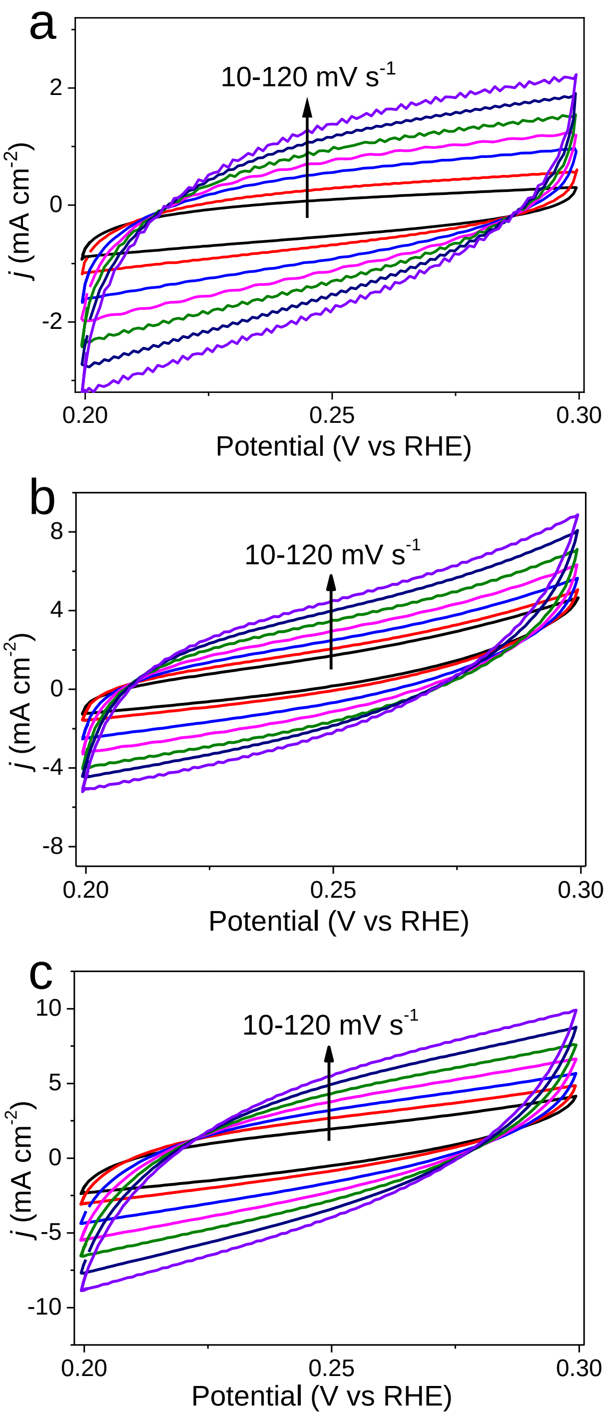

Fig. S4 CV curves of (a) Ni_2P, (b) Fe-Ni_2P and (c) Fe-Ni_2P/CeO_2.

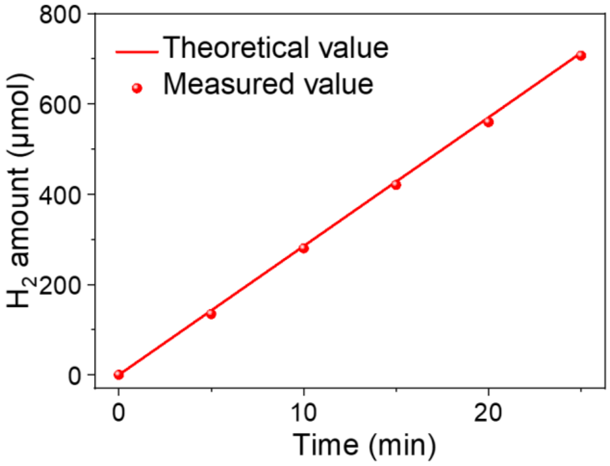

Fig. S5 Experimental and theoretical calculated amount of H_2 over the Fe-Ni_2P/CeO_2.

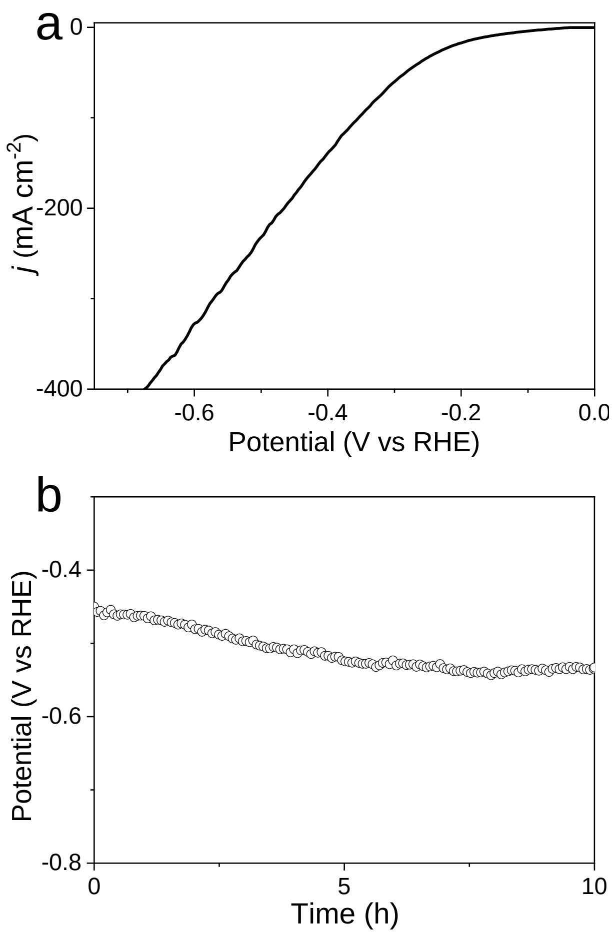

Fig. S6 (a) Polarization curves (b) durability test of Fe-Ni_2P/CeO_2 in 1M PBS.

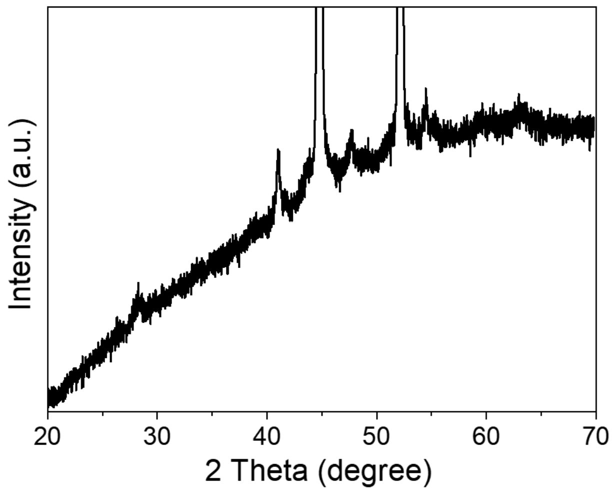

Fig. S7 XRD pattern of Fe-Ni_2P/CeO_2 after the HER measurement.

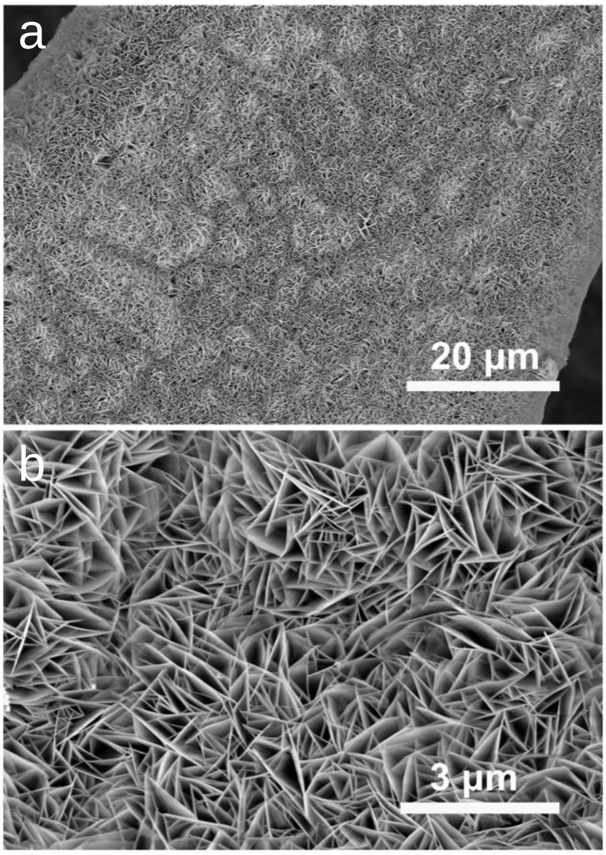

Fig. S8 SEM images of Fe-Ni_2P/CeO_2 after the HER measurement.

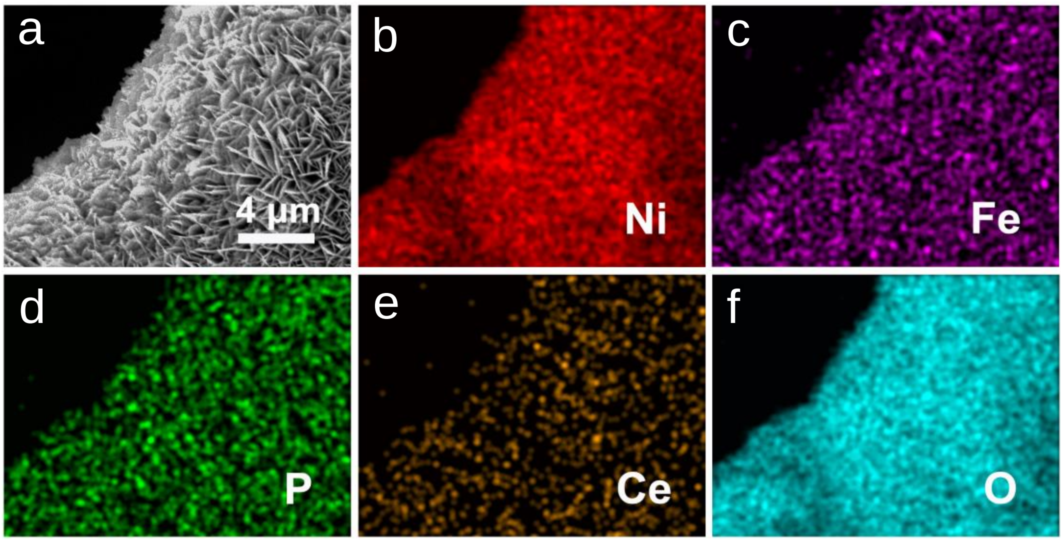

Fig. S9 SEM and corresponding elemental mapping images of Fe-Ni_2P/CeO_2 after the HER measurement.

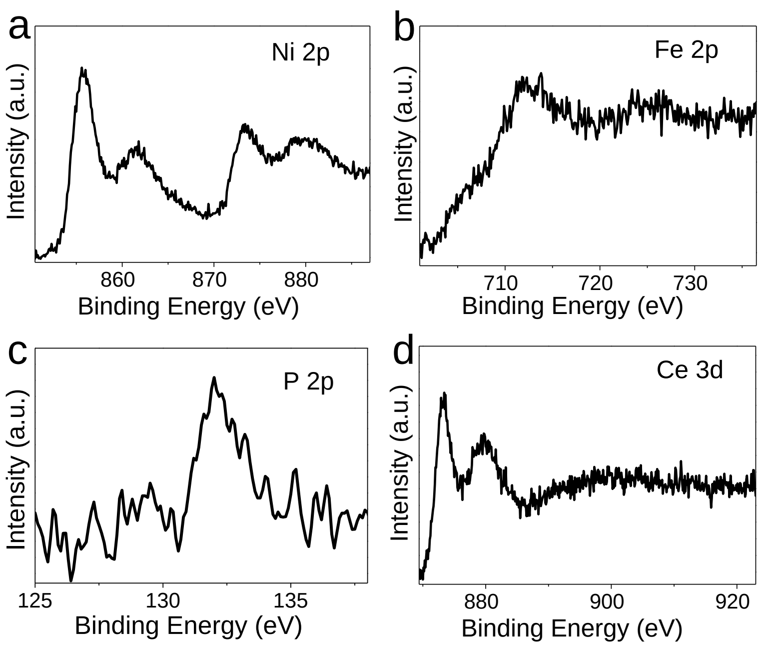

Fig. S10 XPS spectra of Fe-Ni_2P/CeO_2 after the HER measurement.

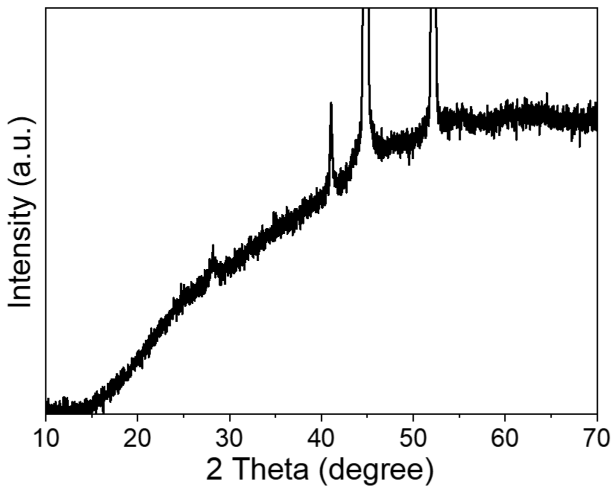

Fig. S11 XRD pattern of Fe-Ni_2P/CeO_2 after the HzOR measurement.

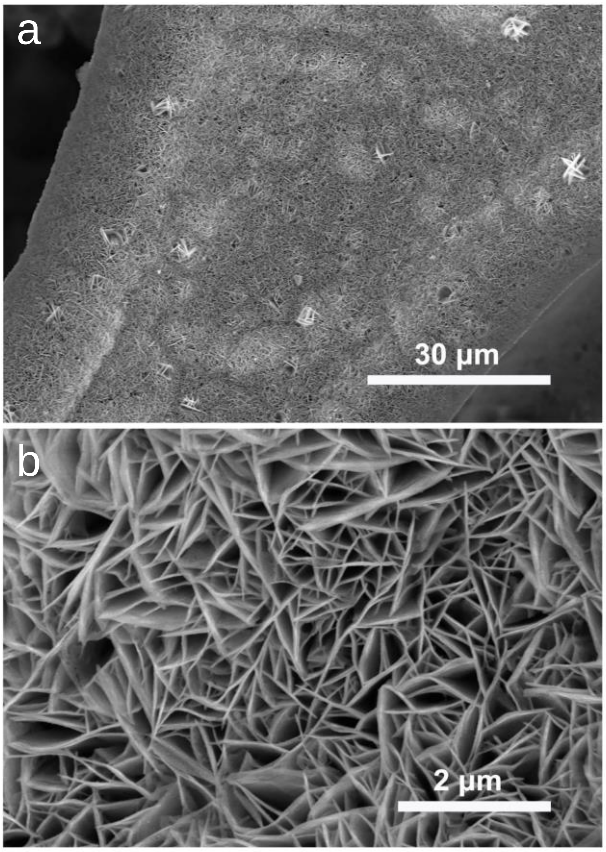

Fig. S12 SEM images of Fe-Ni_2P/CeO_2 after the HzOR measurement.

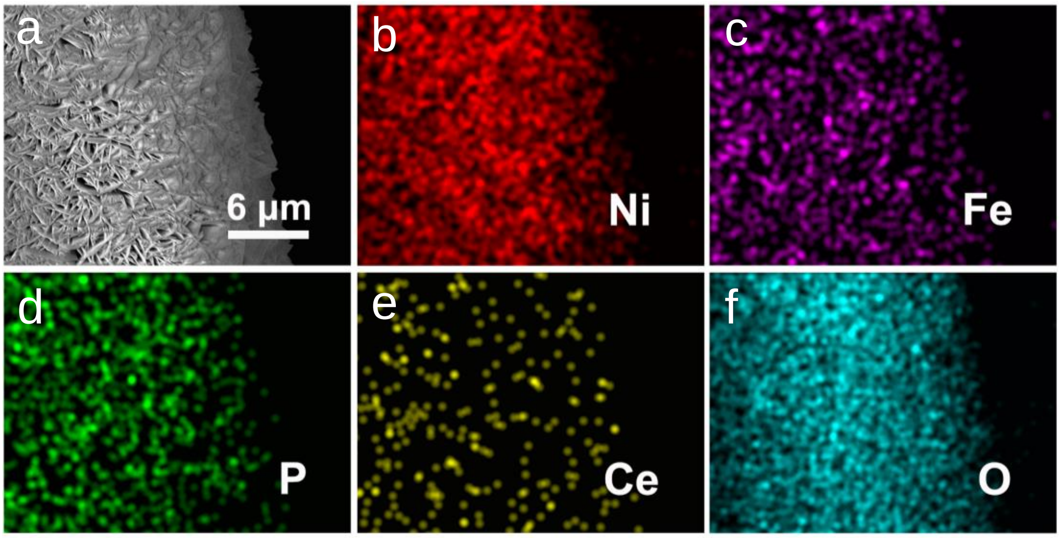

Fig. S13 SEM and corresponding elemental mapping images of Fe-Ni_2P/CeO_2 after the HzOR measurement.

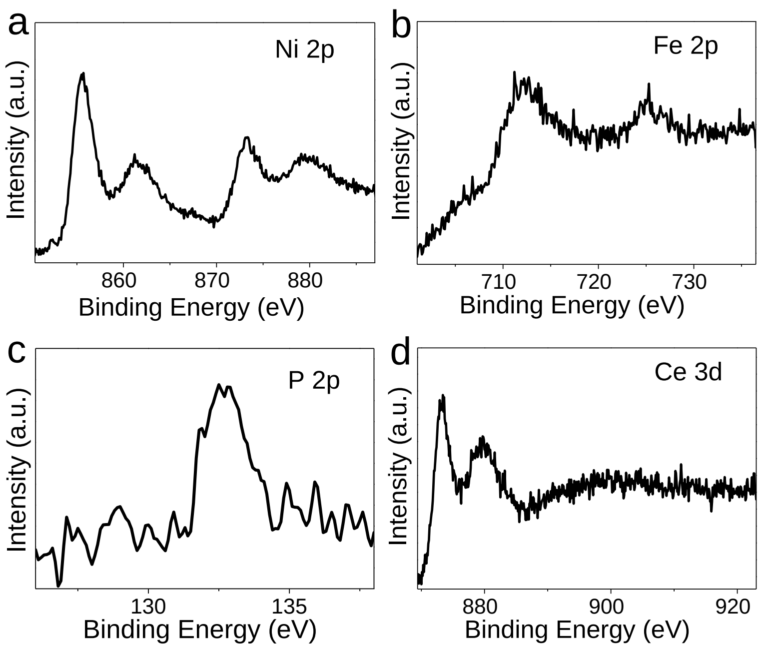

Fig. S14 XPS spectra of Fe-Ni_2P/CeO_2 after the HzOR measurement.

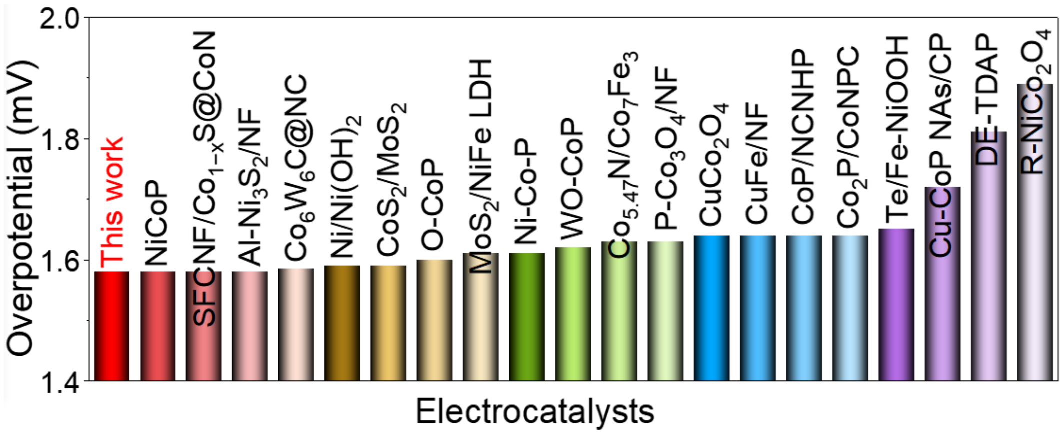

Fig. S15 Comparison of OWS performances of Fe-Ni_2P/CeO_2 with developed catalysts.

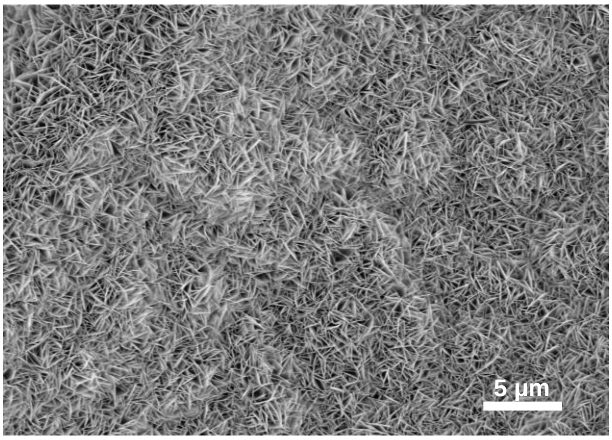

Fig. S16 SEM image of Fe-Ni_2P/CeO_2 at the cathode after the OHzS test.

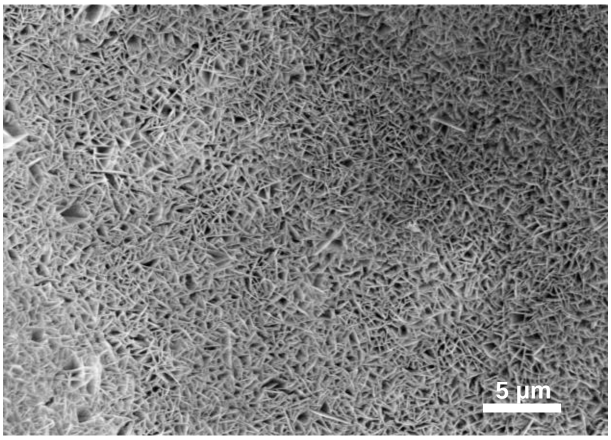

Fig. S17 SEM image of Fe-Ni_2P/CeO_2 at the anode after the OHzS test.

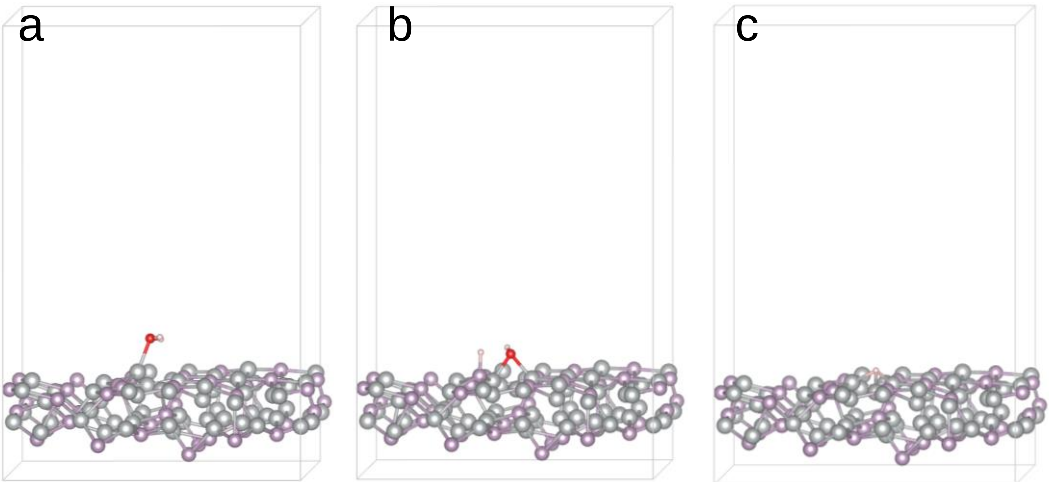

Fig. S18 The structural model of adsorption and dissociation of water molecule and H* on the Ni_2P.

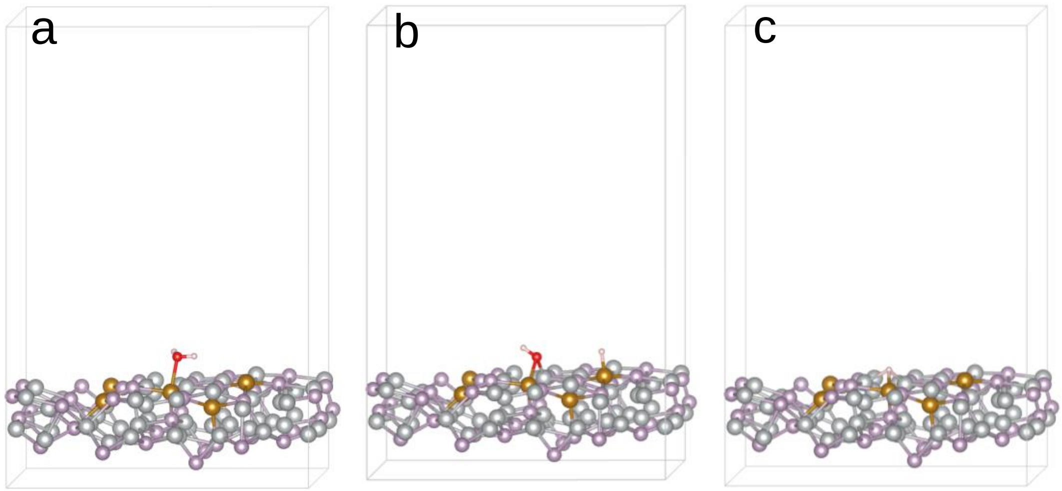

Fig. S19 The structural model of adsorption and dissociation of water molecule and H* on the Fe-Ni_2P.

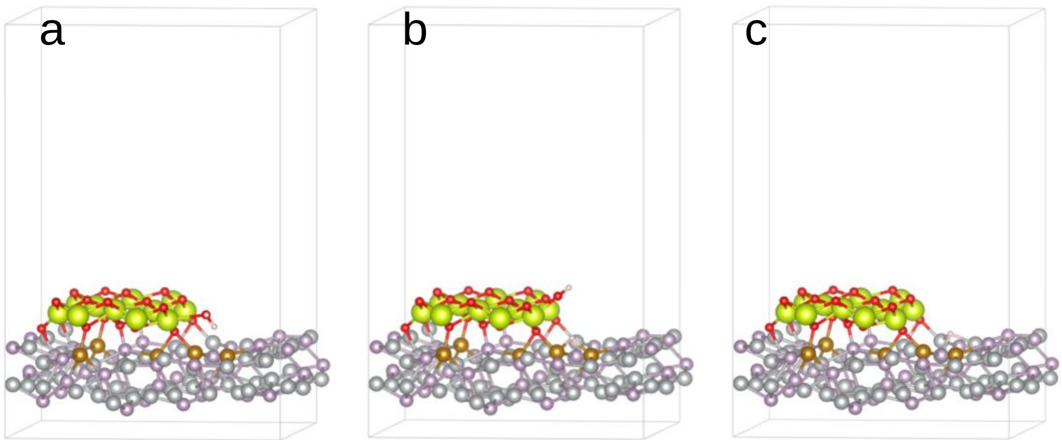

Fig. S20 The structural model of adsorption and dissociation of water molecule and H* on the Fe-Ni_2P/CeO_2.

Table S1 The comparison of HER performance of Fe-Ni_2P/CeO_2 with recently reported materials.

Catalysts	Overpotential (mV)	Reference
Fe-Ni_2P/CeO_2	**67**	**This work**
NiMoP/NF	68	6
CoP·5CoMoP	72	7
Co-P@PC	76	8
NiFeP-NW	83	9
PMo_{12}-MA	89	10
MoS_2 ML	90	11
Mo,Fe-$NiCoP_x$/NF	99	12
NiCoN/C	103	13
Ni-N_3	103	14
Fe(OH)x@Cu-MOF	112	15
NiFeP/SG	115	15
Mo_2CT_x/2H-MoS_2	119	17
$Ti_3CN(OH)_x$@MoS_2	120	18
MC-M_2C/PNCDs	121	19
c-NiP_2/m-NiP_2	134	20
P-$CoNi_2S_4$	135	21
MoP/NPG	150	22
Mo-$Co_{0.85}Se_{VSe}$/NC	151	23
Mn-Ni_2P/NiFe LDH	184	24
H-TaS_2	230	25

Table S2 The comparison of HzOR performance of Fe-Ni_2P/CeO_2 with recently reported materials.

Catalysts	Potential (mV)	Reference
Fe-Ni_2P/CeO_2	**-117**	**This work**
Ni_3N-Co_3N	-88	26
N-fcc-Rh	-81	27
WS_2/Ru	-74	28
CoP/Co	-69	29
Ru_1-NiCoP	-60	30
PW-Co_3N	-55	31
Cu1Co_2–Ni_2P	-52	32
Ru-Cu_2O/CF	-41	33
$FeWO_4$-WO_3/NF	-34	34
Ni NCNAs	-26	25
Ni-C HNSA	-20	36
NiMo/Ni_2P	-17	37
$Ni(OH)_2/Ni_2P$/NF	-14	38
Rh_2P uNSs	-10	39
FHNNP/NF	-8	40
Mo-Ni_3N/Ni/NF	-0.3	41
Cu1Ni_2-N	0.5	42
Co/BNC	95	43
Ni_2P/Co_2P	230	44
Ir-SA/NC	390	45

Table S3 The comparison of OWS performance of Fe-Ni_2P/CeO_2 with recently reported materials.

Catalysts	Voltage (V)	Reference
Fe-Ni_2P/CeO_2	1.582	This work
NiCoP	1.58	46
SFCNF/$Co_{1-x}S$@CoN	1.58	47
Al-Ni_3S_2/NF	1.58	48
Co_6W_6C@NC	1.585	49
Ni/$Ni(OH)_2$	1.59	50
CoS_2/MoS_2	1.59	51
O-CoP	1.60	52
MoS_2/NiFe LDH	1.61	53
Ni-Co-P	1.61	54
WO-CoP	1.62	55
$Co_{5.47}N/Co_7Fe_3$	1.63	56
P-Co_3O_4/NF	1.63	57
$CuCo_2O_4$	1.64	58
CuFe/NF	1.64	59
CoP/NCNHP	1.64	60
R-$NiCo_2O_4$	1.64	61
Te/Fe-NiOOH	1.65	62
Mo-CoP	1.70	63
Cu-CoP NAs/CP	1.72	64
DE-TDAP	1.81	65

Table S4 The comparison of OHZS performance of Fe-Ni_2P/CeO_2 with recently reported materials.

Catalysts	Voltage (V)	Reference
Fe-Ni_2P/CeO_2	51	This work
Mo-Ni_3N/Ni/NF	55	41
Rh/RhO_x	68	66
Ni_3N-Co_3N	71	26
a-RhPb NFs	95	67
Co/BNC	103	43
Rh_2S_3/NC	108	68
Mn-CoS_2	111	69
RhIr MNs	130	70
Mo-Ni_2P_v@MNF	133	71
RuFe-Ni_2P@NF	140	72
$Co(OH)_2/MoS_2$/CC	142	73
Cu1Co_2–Ni_2P	160	32
$CoSe_2$	164	74
FHNNP/NF	220	40
Cu1Ni_2-N	240	42
PW-Co_3N	358	31
Rh_2P uNSs	377	39
Ir-SA/NC	390	45
Mn-SA/BNC	410	75
Cu_1Pd_3/C	505	76

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