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Supporting Information

Ultrathin Vanadium Hydroxide Nanosheets Assembled on the Surface of Ni-Fe Layered Hydroxides as a Hierarchical Catalyst for Oxygen Evolution Reaction

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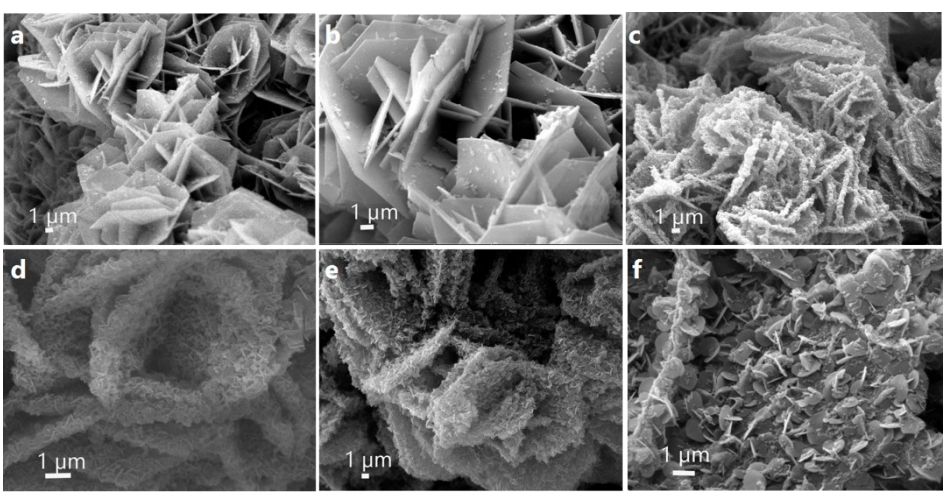

Fig. S1. (a-b) SEM images of NiFe LDHs/NF; (c-d) SEM images of NFV NSs-8h;

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and (e-f) SEM images of NiFe NFV NSs-12h.

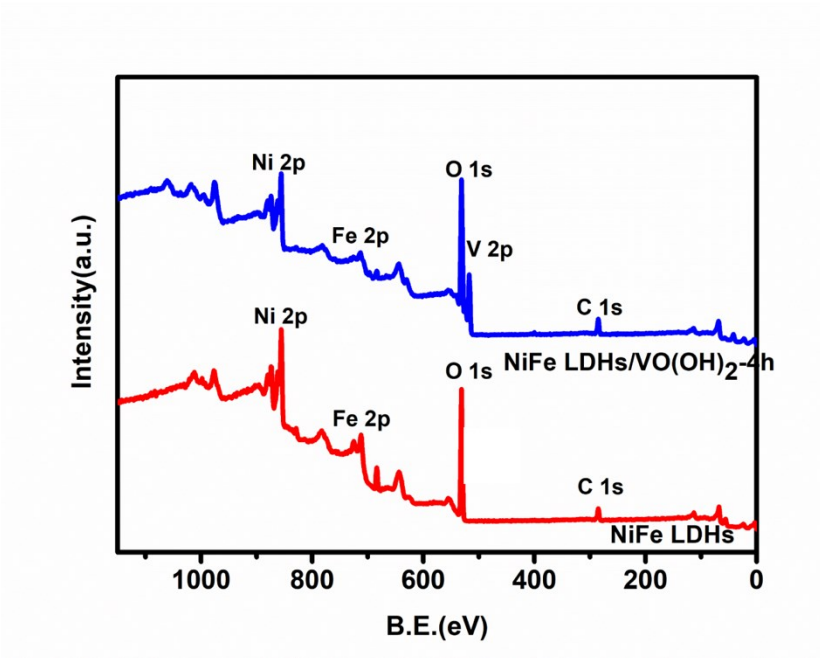

Fig. S2. XPS survey spectra of NFV NSs-4h and NiFe LDHs/NF.

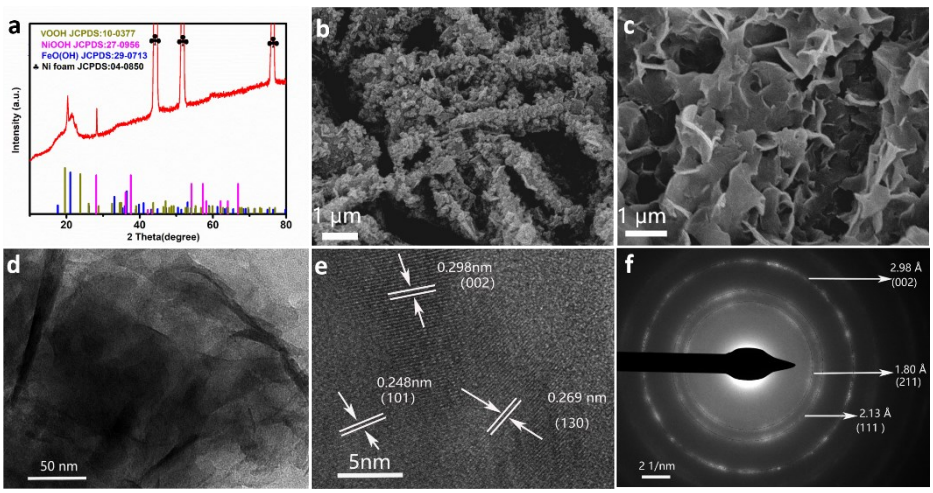

Fig. S3. (a) XRD patterns of NFV NSs-4h after long-term OER process; (b-c) SEM images; (d) TEM image, (e) HR-TEM image and (f) SAED patterns of NFV NSs-4h after OER.

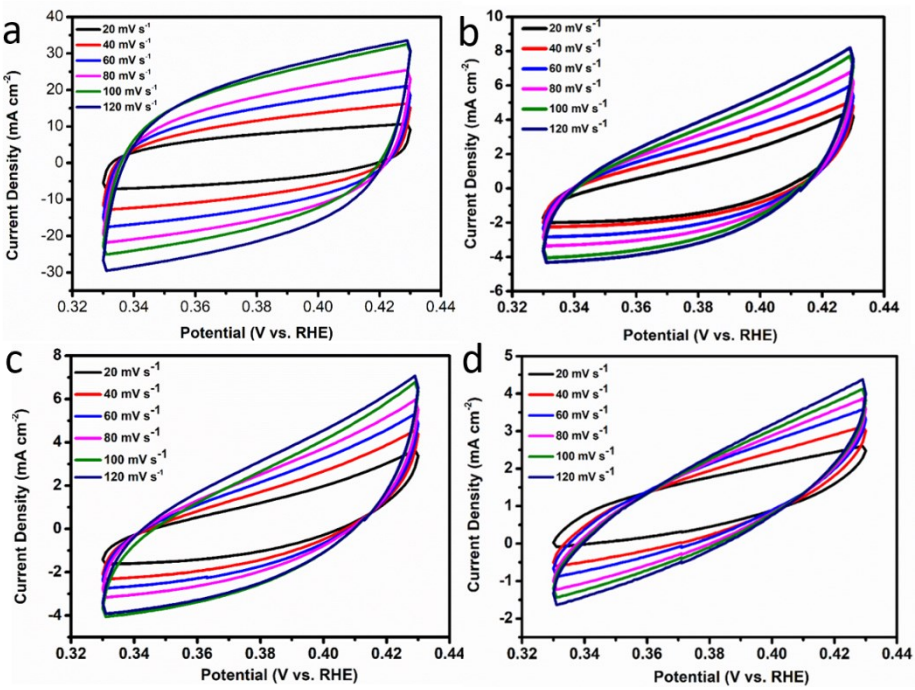

Fig. S4. CV curves of (a) NFV NSs-4h, (b) NFV NSs-8h, (c) NFV NSs-12h, (d) NiFe LDHs/NF.

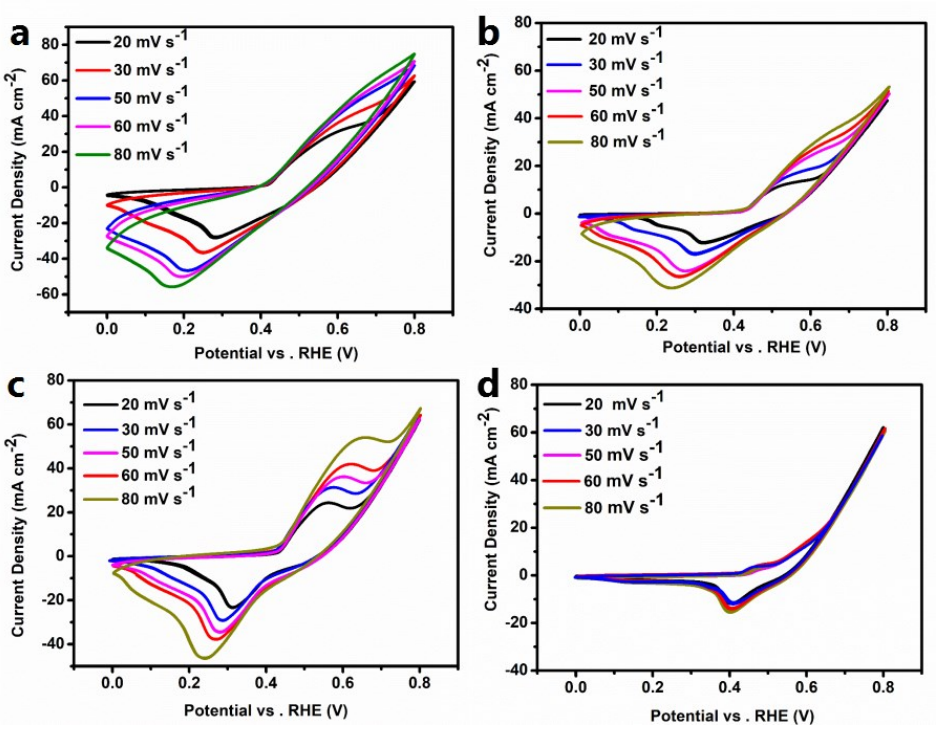

Fig. S5. CVs for NiFe-LDHs and NFV NSs-xh (4,8,12) in faradic capacitance current range at various scan rates: (a) NFV NSs-4h, (b) NFV NSs-8h, (c) NFV NSs-12h, (d) NiFe LDHs/NF.

Table S1. Comparison of the OER performance for the obtained materials in this work with other state-of-the-art OER electrocatalysts.

Catalyst	Electrolyte	$\eta_{j=100}$ (mV)	$\eta_{j=200}$ (mV)	Tafel slope [mV dec^{-1}]	Reference
NFV NSs-4h	1 M KOH	280	300	65	This work
NFV NSs-8h	1 M KOH	300	320	84	This work
NFV NSs-12h	1 M KOH	320	350	112	This work
NiFe LDHs	1 M KOH	310	330	96	This work
CS-NiFeCu	1 M KOH	~210	~240	33	[1]
$NiFe_2O_4$	1 M KOH	~210	/	46.4	[2]
NiCoFe-LDH HP	1 M KOH	332	~340	56	[3]
0.1Fe-CoNiO/NF	1 M KOH	~280	/	36.8	[4]
Fe_3O_4-FeSe/$CoSe_2$	1 M KOH	~350	~370	68.7	[5]
$Co_{2.4}Ni_{0.6}Ge_2O_5(OH)_4$	1 M KOH	349	~370	59.8	[6]
Cu oxide micro/nano-structures	1 M KOH	~350	~370	63	[7]
NF@NC-$CoFe_2O_4$/C NRAs	1 M KOH	~290	~310	45	[8]

Experimental

1.1 Materials and chemicals

Ammonia fluoride (NH_4F, 96.0%), nickel nitrate hexahydrate ($Ni(NO_3)_2.6H_2O$, 99.0%) and anhydrous ethanal (CH_3CH_2OH, 99.7%) were purchased from Sinopharm Chemical Reagent Co., Ltd. Vanadium trichloride (VCl_3, 99.0%) and urea (CH_4N_2O, 99%) were obtained from Aladdin. Iron chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, 99.0%) was supplied by Kelong Chemical Reagent. Nickel foam (NF) (thickness = 0.5 mm) was provided by Guangdong Candlelight New Energy Technology Co., Ltd.

1.2 Synthesis of NiFe LDHs/NF

The NiFe LDHs/NF was fabricated by a one-step hydrothermal method. Prior to the synthesis, NF (2.5 cm x 4 cm) was pretreated ultrasonically in HCl (3.0 M) for 20 minutes to remove the impurities, and then washed it by deionized water and ethanol for several times until pH ~ 7. In a typical process, $Ni(NO_3)_2 \cdot 6H_2O$ (1 mmol), $FeCl_3 \cdot 6H_2O$ (2 mmol), NH_4F(40 mmol) and CH_4N_2O were dissolved in 40 mL deionized water to form a clear solution and then transferred into a 50 mL Teflon-lined

autoclave. A piece of cleaned NF was immersed into the mixture solution. The autoclave was sealed and maintained at 120 ^{o}C for 6 h. After cooled down to room temperature, the NiFe LDHs/NF was washed with deionized water several times and dried at 60 ^{o}C for 8 hours.

1.3 Synthesis of NiFe LDH/$VO(OH)_2$-xh

VCl_3 (1.6 mmol) and CH_4N_2O (0.3 g) were dissolved in 40 mL deionized water to form a homogeneous solution. Then, the solution was transferred into a 50 mL Teflon-lined autoclave and the NiFe LDHs/NF was completely immersed into the solution. The autoclave was sealed and heated at 120 ^{o}C for x hours (x = 4, 8, 12). After cooled to room temperature, the NF with catalyst was washed with deionized water several times and dried at 60 ^{o}C for 8 hours. The as-obtained catalysts are denoted as NiFe LDH/$VO(OH)_2$-xh (NFV NSs-xh; x = 4, 8, 12).

1.4 Materials characterization

The morphology of the samples was observed by scanning electron microscope (SEM, S-4800, Japan) and transmission electron microscopy (TEM, G2F20, USA). The crystal structure and chemical composition of the samples was analyzed by X-ray diffraction (XRD, Smart Lab) and X-ray photoelectron spectroscopy (XPS, PHI 5000), respectively.

1.5 Electrochemical measurements

The electrochemical properties of the materials were evaluated in a three-electrode system on a CHI-660E electrochemical workstation (Chenhua instrument co., LTD., Shanghai) at room temperature. In a standard three-electrode system, the NF (0.5 cm × 0.5 cm) with different samples was directly used as working electrode, then a Hg/HgO electrode and a graphite rod were acted as the reference electrode and the counter electrode, respectively. All measured potentials were calibrated to the reversible hydrogen electrode (RHE) based on the Nernst equation: $E_{RHE} = E_{Hg/HgO} + 0.059 \times pH + 0.098$. The steady-state linear sweep voltammetry (LSV) curves were obtained at a scan rate of 2 mV s^{-1} in 1.0 M KOH solution. The measurements of electrochemical impedance spectroscopies (EIS) were conducted at a frequency range from 10 KHz to 100 mHz by applying an alternating current (AC) voltage with 10 mV amplitude. The chronoamperometric test (40 h) of NFV NSs-4h for OER was carried out at a current density of 100 mA cm^{-2} and cyclic voltammetry (CV) tests of 1000 cycles were conducted at a scan rate of 100 mV s^{-1} to investigate the durability of the sample. In

order to determine the electrochemical active surface area (ECSA), the double layer capacitance (C_{dl}) of the electrode can be obtianed by carrying out the CV measurement and the ECSA can be calculated by C_{dl} using the formula: ECSA = C_{dl} / C_s (C_s is assuming as 0.040 mF cm^{-2}).[9]

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