

Supporting Information for

Prompt Electrodeposition of Ni Nanodots on Ni Foam to Construct a High-Performance Water Splitting Electrode: Efficient, Scalable, and Recyclable

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Supplementary Figures and Table

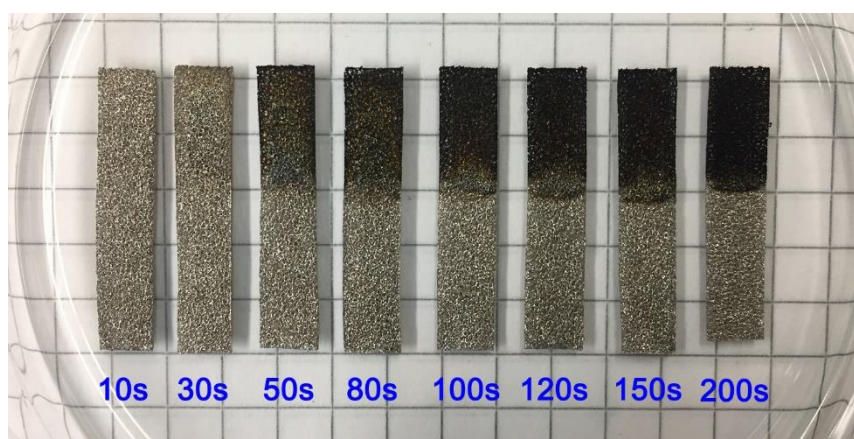


Fig. S1 Digital photograph of NiO/NiNDs@NF with different electrodeposition time. During this process, an apparent color change of NF was observed. The color gradually changed from silver-white to black because of the increase in deposited NiNDs

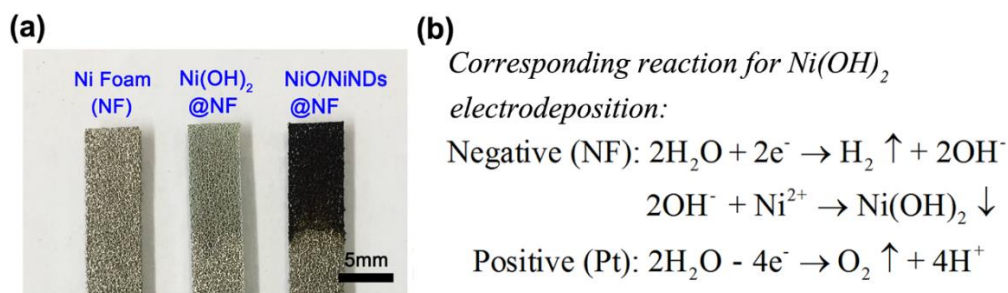


Fig. S2 a Digital photograph of Ni foam, Ni(OH)₂@NF, and NiO/NiNDs@NF. **b** Corresponding chemical reaction for Ni(OH)₂ electrodeposition in aqueous solution

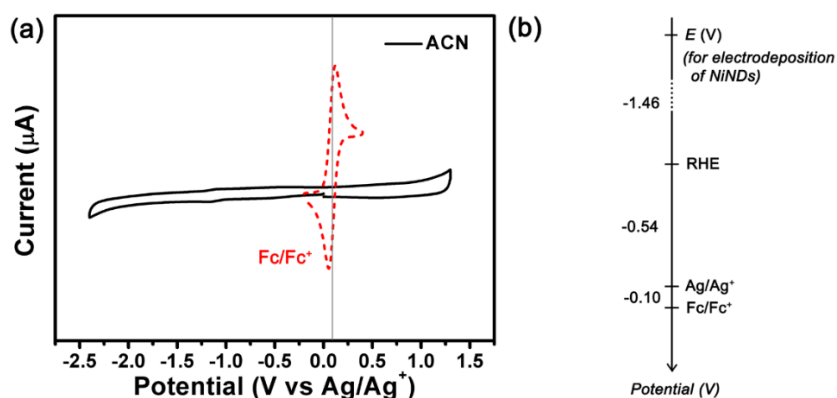


Fig. S3 a Potential window of ACN. CV measurement was performed using a standard three-electrode system controlled by a GAMRY 11100 electrochemistry workstation. GCE was used as the working electrode, Pt wire was used as the counter electrode and Ag/Ag⁺ was used as the reference electrode. The scan rate was 50 mV s⁻¹. **b** The reference for electrodeposition was calibrated against and converted to the reversible hydrogen electrode (RHE) through the potential of Fc/Fc⁺ [S1]

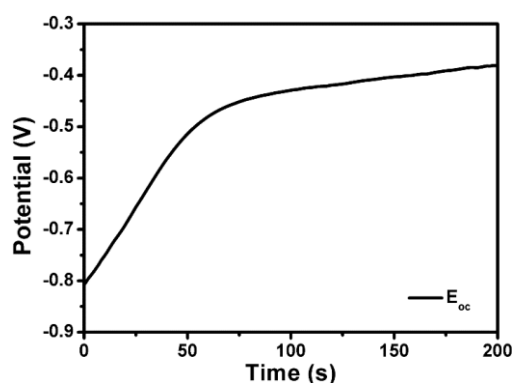


Fig. S4 Open circuit potential of a freshly obtained NiNDs@NF electrode, which shows a high negative open circuit potential (E_{oc}). This potential dropped drastically after the first dozens of seconds because of the quick oxidation of external NiNDs ($\text{Ni}^0 \rightarrow \text{Ni}^{2+}$). The decreasing in this value becomes gentle with further oxidation of the internal NiNDs

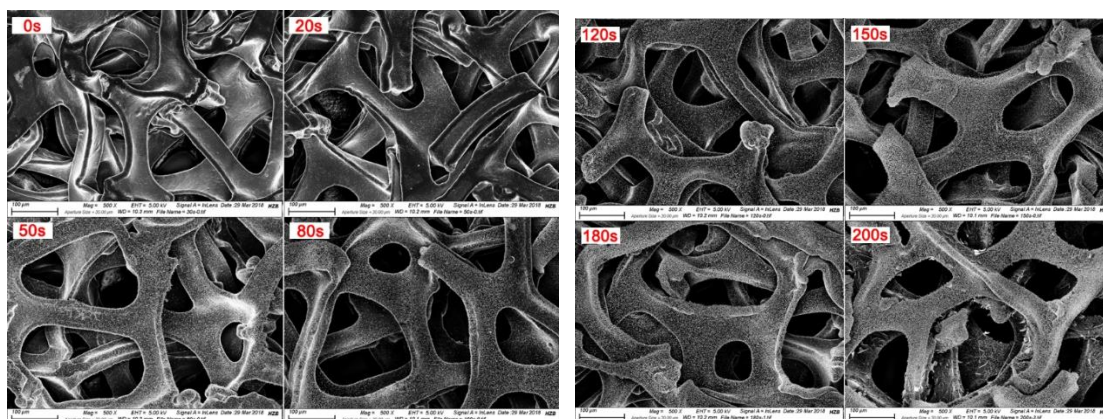


Fig. S5 Large-scale SEM images of NiO/NiNDs@NF with different electrodeposition time

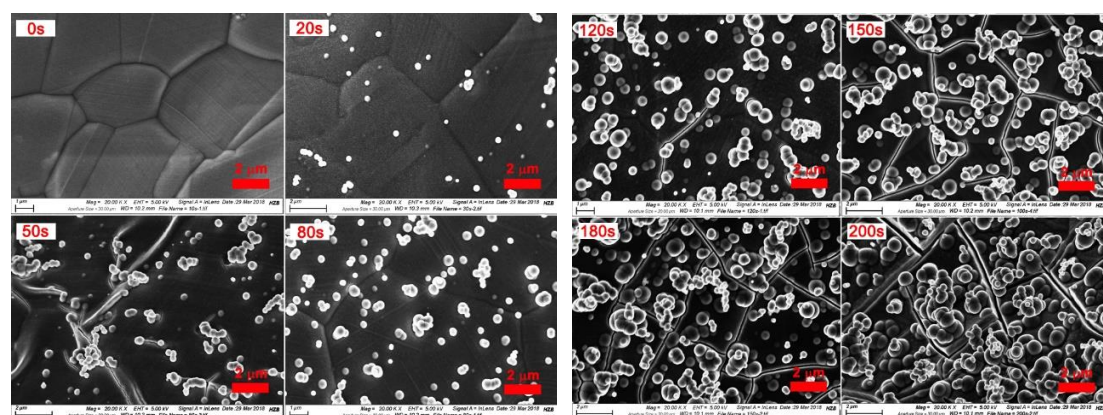


Fig. S6 Low-scale SEM images of NiO/NiNDs@NF with different electrodeposition time. Integrated spherical nodules of NiND clusters with different sizes formed gradually on NF

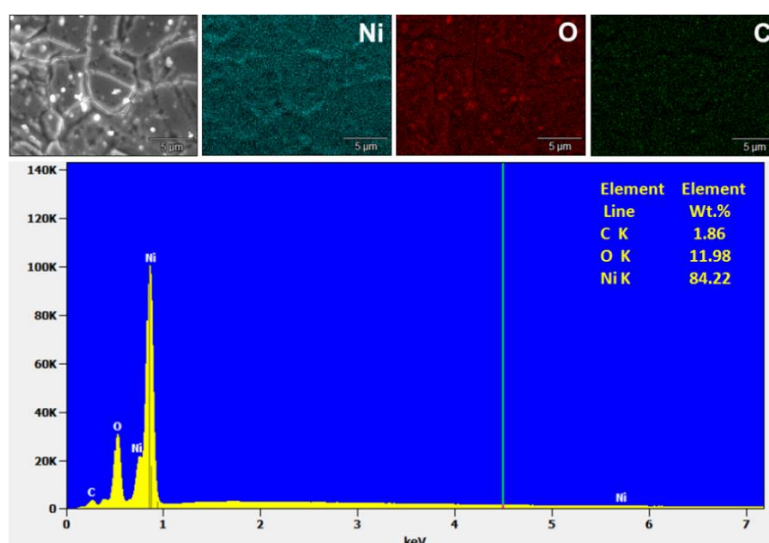


Fig. S7 EDX spectrum and mapping of Ni, O, and C element of the NiO/NiNDs@NF

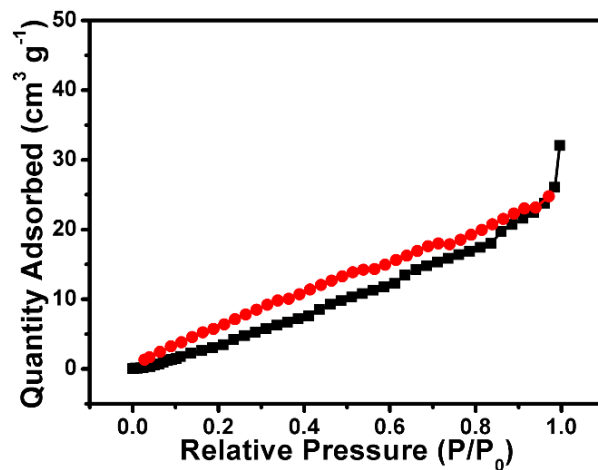


Fig. S8 Nitrogen sorption isotherms of the NiO/NiNDs samples

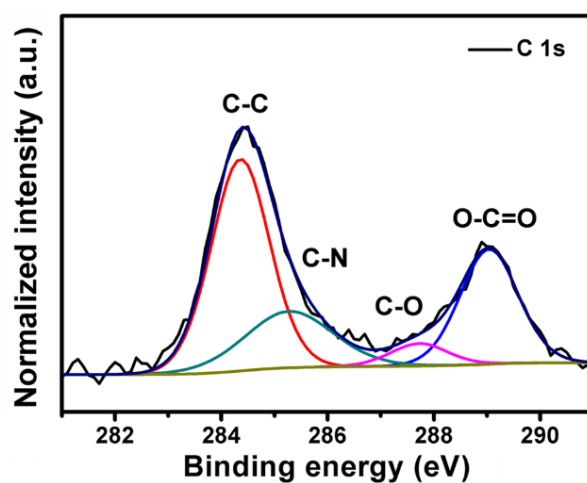


Fig. S9 High-resolution XPS C 1s spectra

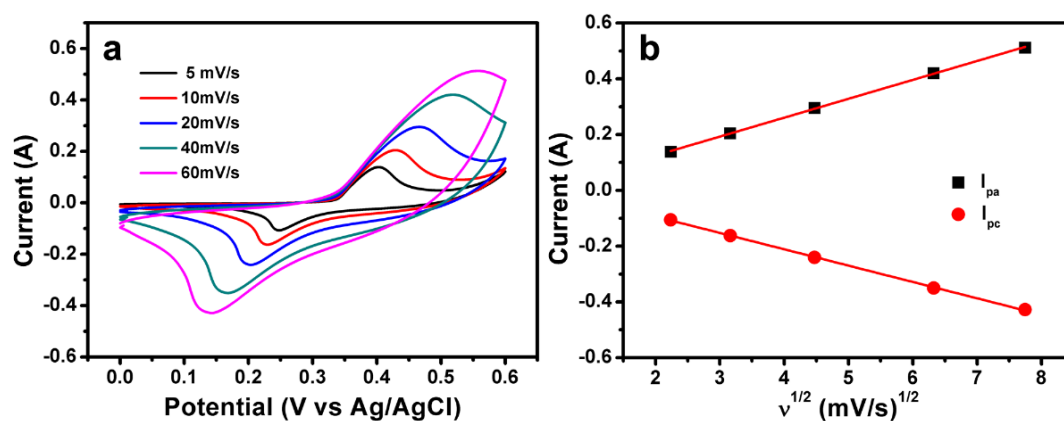


Fig. S10 a CV curves of the NiO/NiNDs@NF electrode with different scan rates. **b** Anodic and cathodic peak current densities obtained from the CV curves and plotted as a function of the square root of the scan rates

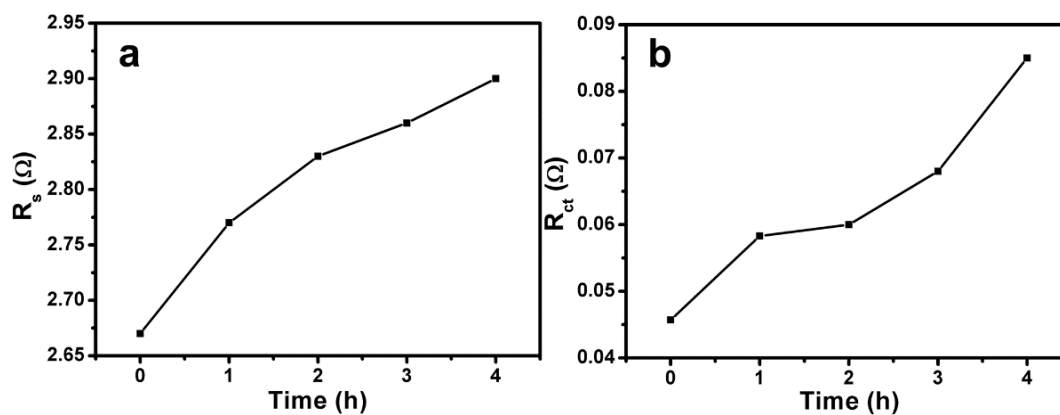


Fig. S11 **a** R_s and **b** R_{ct} of EIS spectra of NiO/NiNDs@NF electrodes as a function of the heating time in a 200 °C oven

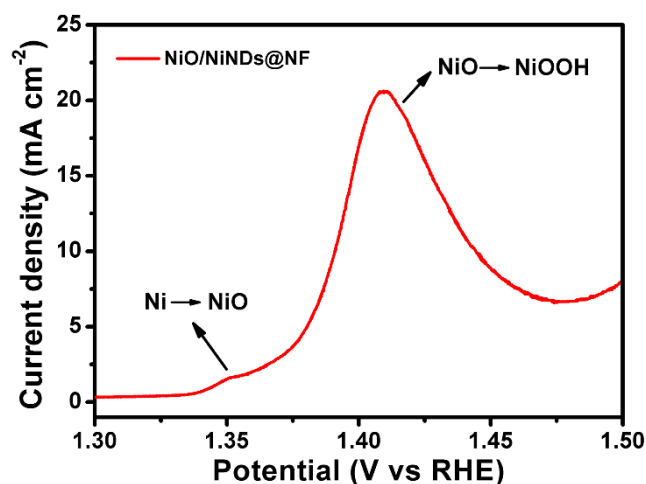


Fig. S12 Magnified LSV curve of NiO/NiNDs for OER

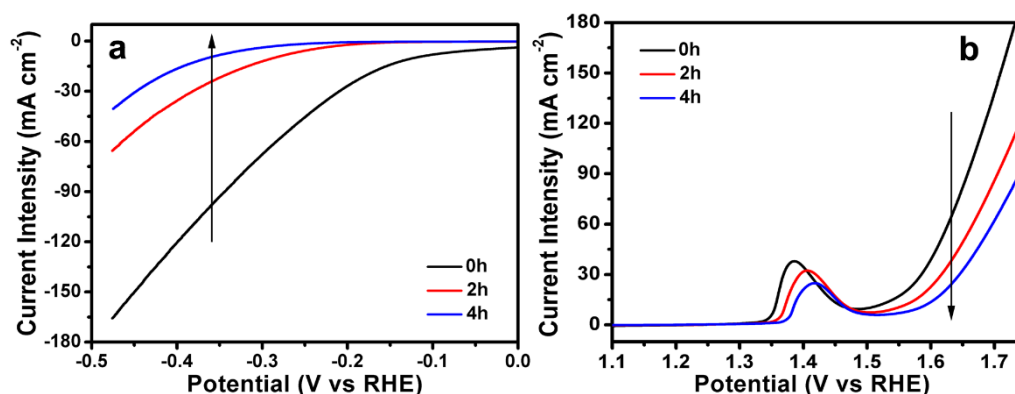


Fig. S13 Electrocatalytic performance of the 120 s' deposited NiO/NiNDs@NF electrodes with different heating time (0, 2, and 4 h) at 200 °C. Linear sweep voltammetry of the corresponding NiO/NiNDs@NF electrodes in 1M KOH at a scan rate of 5 mV s⁻¹ for **a** HER and **b** OER

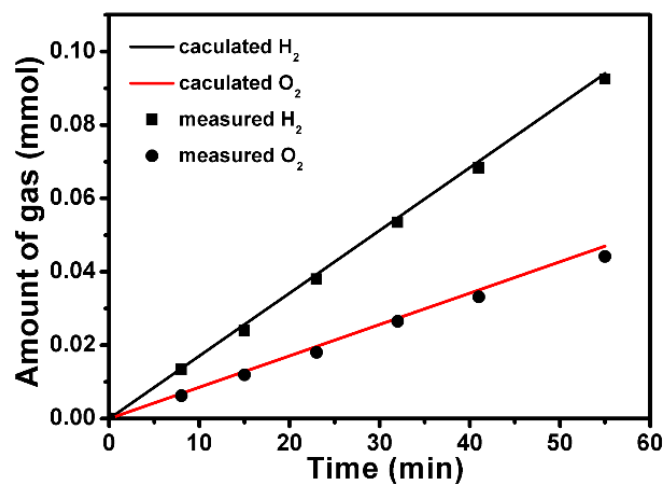


Fig. S14 Amount of gas theoretically calculated and experimentally measured versus time for NiO/NiNDs in 1.0 M KOH

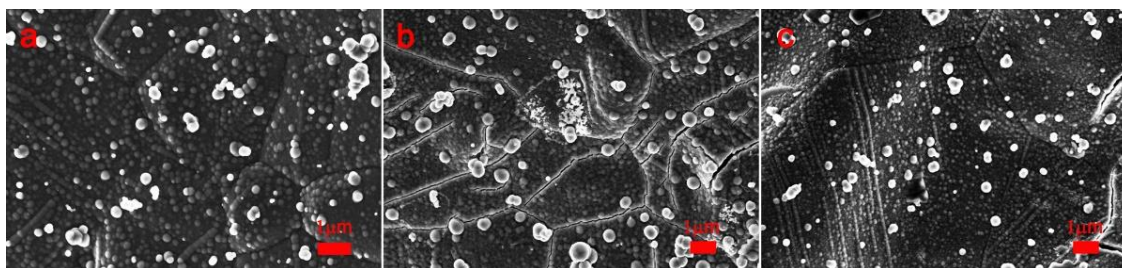


Fig. S15 Morphologies of NiO/NiNDs@NF **a** before and **b** after long-term HER and **c** OER tests

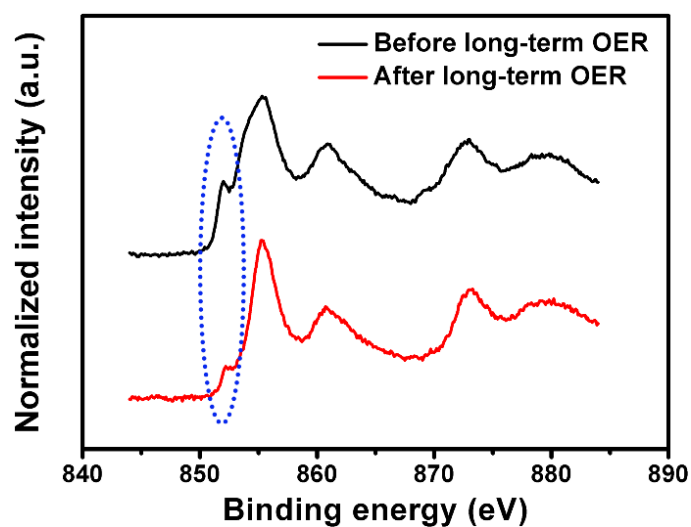


Fig. S16 High-resolution XPS Ni 2p spectra before and after long-term OER measurement

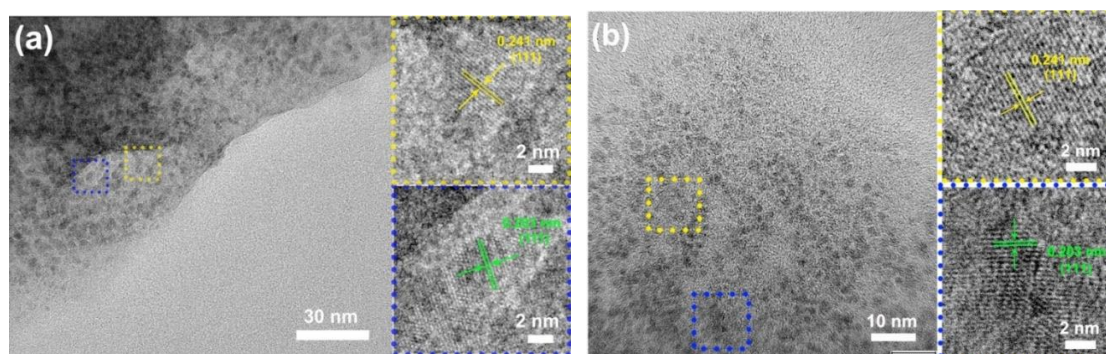


Fig. S17 HRTEM images of NiO/NiNDs@NF after long-term **a** HER and **b** OER tests. The lattice fringe spacing of 0.241 nm corresponds to the (111) plane of NiO (JCPDS (Joint Committee on Powder Diffraction Standards) No. 47-1049). Another interplanar spacing of 0.203 nm matches well with the d_{111} spacing of metal Ni (JCPDS No. 65-2865), revealing that the NiND in the NiO/NiND composites are stable during long-term HER and OER processes

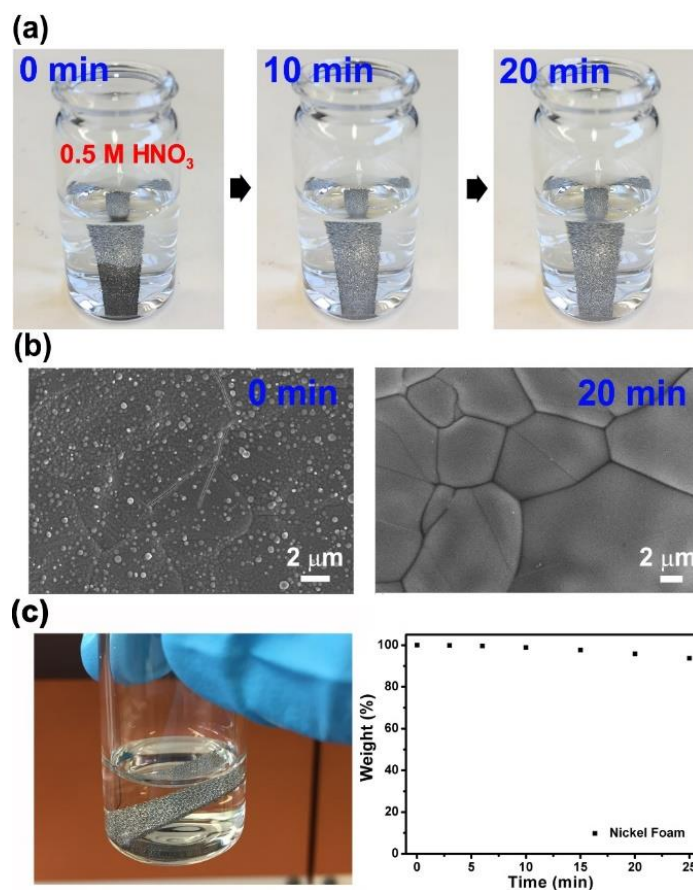


Fig. S18 a Photographs of NiO/NiNDs@NF immersed in 0.5 M HNO₃ solution. **b** SEM images of NiO/NiNDs@NF before (0 min) and after (20 min) treating with 0.5 M HNO₃ solution. **c** Photograph of NF immersed in 0.5 M HNO₃ solution and the change of weight with respect to time

Table S1 Comparison of the overpotential behavior of modified NF

Catalyst	Water electrolysis test	Electrolyte Solution	Current Density (j ; mA cm ⁻²)	Overpotential at the corresponding j (mV)	References
NiO/NiNDs@NF	HER	1 M KOH	10	120	This work
			20	156	
	OER	1 M KOH	20	320	
			50	358	
NiS@NF	HER	1 M KOH	20	158	[S2]
	OER	1 M KOH	50	335	
NiFe LDH@NF	HER	1 M KOH	20	251	[S3]
	OER	1 M KOH	50	349	
Ni ₃ S ₂ @NF	HER	1 M KOH	10	223	[S4]
	HER	1 M KOH	20	292	
	OER	1 M KOH	10	260	
NiSe@NF	HER	1 M KOH	10	96	[S5]
	OER	1 M KOH	20	270	
Ni(OH) ₂ @NF	HER	1 M KOH	10	250	[S3]
	OER	1 M KOH	20	ca. 372	
Ni ₃ N@NF	HER	1 M KOH	10	121	[S6]
			20	177	

Supplementary References

[S1] C.M. Cardona, W. Li, A.E. Kaifer, D. Stockdale, G.C. Bazan, Electrochemical considerations for determining absolute frontier orbital energy levels of conjugated polymers for solar cell applications. *Adv. Mater.* **23**(20), 2367-2371 (2011).
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[S3] J. Luo, J.H. Im, M.T. Mayer, M. Schreier, M.K. Nazeeruddin et al., Water photolysis at 12.3% efficiency via perovskite photovoltaics and earth-abundant catalysts. *Science* **345**(6204), 1593-1596 (2014).
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[S6] Z. Xing, Q. Li, D. Wang, X. Yang, X. Sun, Self-supported nickel nitride as an efficient high-performance three-dimensional cathode for the alkaline hydrogen evolution reaction. *Electrochim. Acta* **191**, 841-845 (2016). <https://doi.org/10.1016/j.electacta.2015.12.174>

Movie S1 Electrodeposition process of NiND clusters

Movie S2 Water electrolysis of large-sized NiO/NiNDs@NF electrodes in 1 M KOH ($S_{electrodes} \sim 70 \text{ cm}^2$; distance between electrodes is $\sim 2 \text{ cm}$). Current density is 13 mA cm^{-2}