

## Supplementary Materials

### **An Amorphous FeNiO<sub>x</sub> Thin Film by Anodic Electrodeposition as Electrocatalyst toward Oxygen Evolution Reaction**

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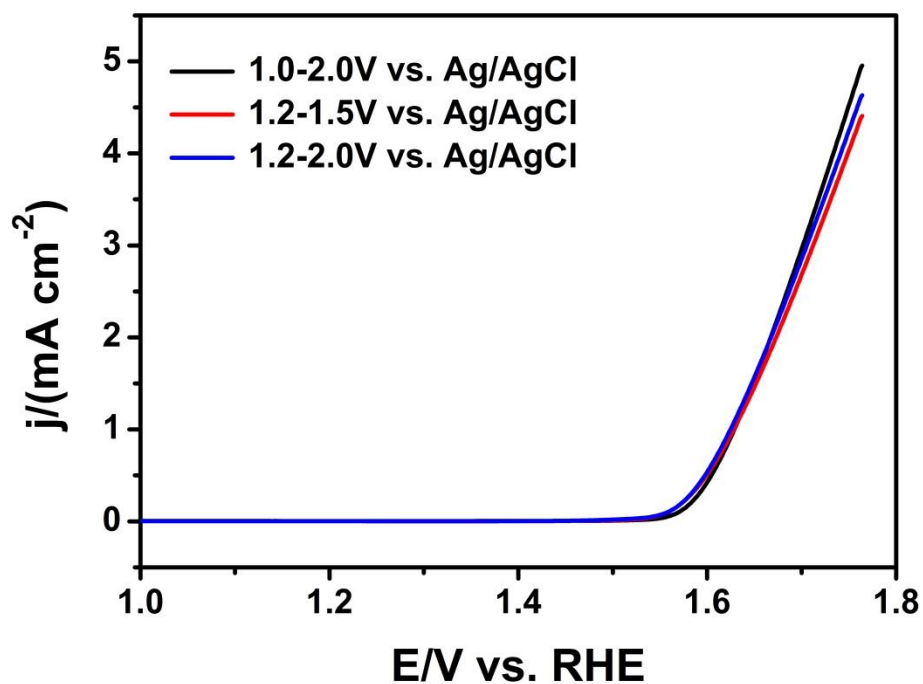
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**TOF calculation:**

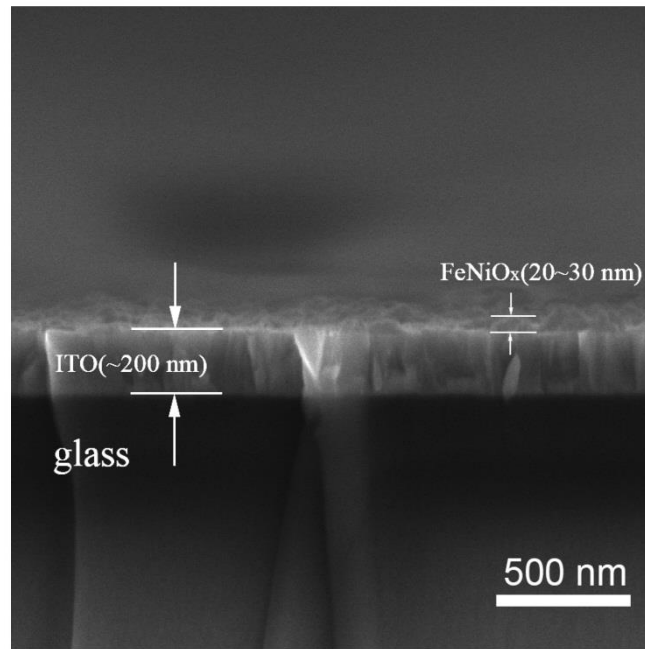
The TOF values were calculated by assuming that every metal atom is involved in the catalysis<sup>1</sup>:

$$\text{TOF} = jS / 4Fn$$

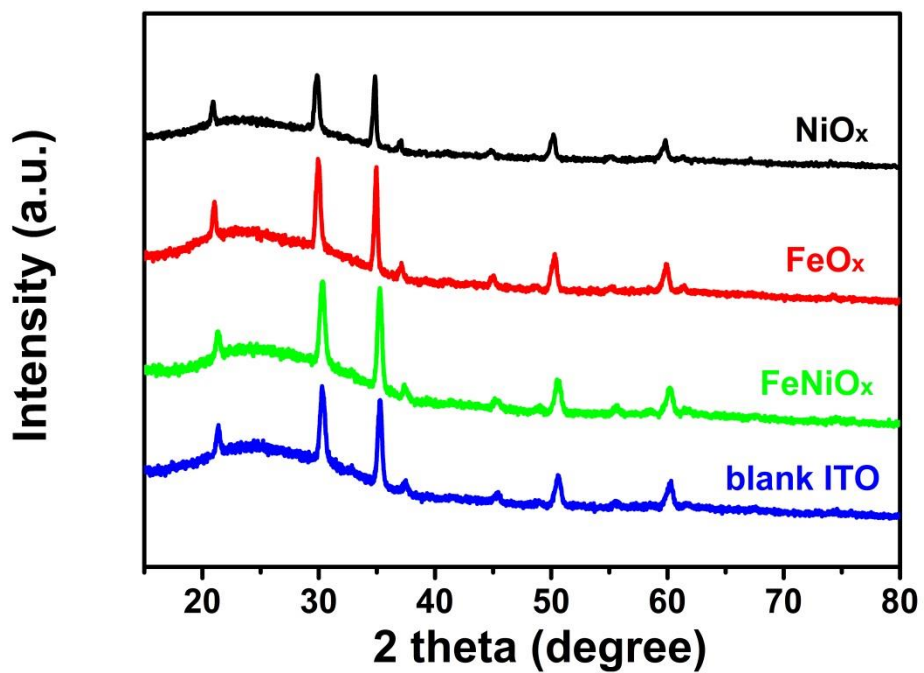
where  $j$  (mA/cm<sup>2</sup>) is the measured current density at  $\eta = 470$  mV,  $S$  is the active surface area of catalyst deposited on ITO, the number 4 means 4 electrons/mol of O<sub>2</sub>,  $F$  is Faraday's constant (96485.3 C/mol), and  $n$  is the moles of coated metal atom on the electrode calculated from mass loading and the molecular weight of the coated catalysts. The TOF of FeNiO<sub>x</sub> is calculated as 0.023 s<sup>-1</sup>, which is much higher than NiO<sub>x</sub> (0.001 s<sup>-1</sup>) and FeO<sub>x</sub> (0.0002 s<sup>-1</sup>).



**Fig. S1** LSV of  $\text{FeNiO}_x$  catalysts via different electrodeposition CV potentials (in the range of 1.0 – 2.0 V, 1.2 – 1.5 V, 1.2 – 2.0 V (all vs. Ag/AgCl), respectively.) Deposition conditions: ITO substrate, 10 mM nickel(II) sulfate, 5 mM iron(III) sulfate, 0.1 M sodium acetate,  $\text{pH} \approx 5.30$ ,  $20 \text{ mV s}^{-1}$ , 75 CVs. All electrochemical measurements were performed in a 0.1 M KOH ( $\text{pH} \approx 13.0$ , scan rate:  $10 \text{ mV s}^{-1}$ , room temperature).



**Fig. S2** The cross-section SEM image of the FeNiO<sub>x</sub>/ITO catalyst. Scale bar corresponds to 500 nm.



**Fig. S3** XRD patterns of NiO<sub>x</sub>/ITO, FeO<sub>x</sub>/ITO, FeNiO<sub>x</sub>/ITO thin films and blank ITO.

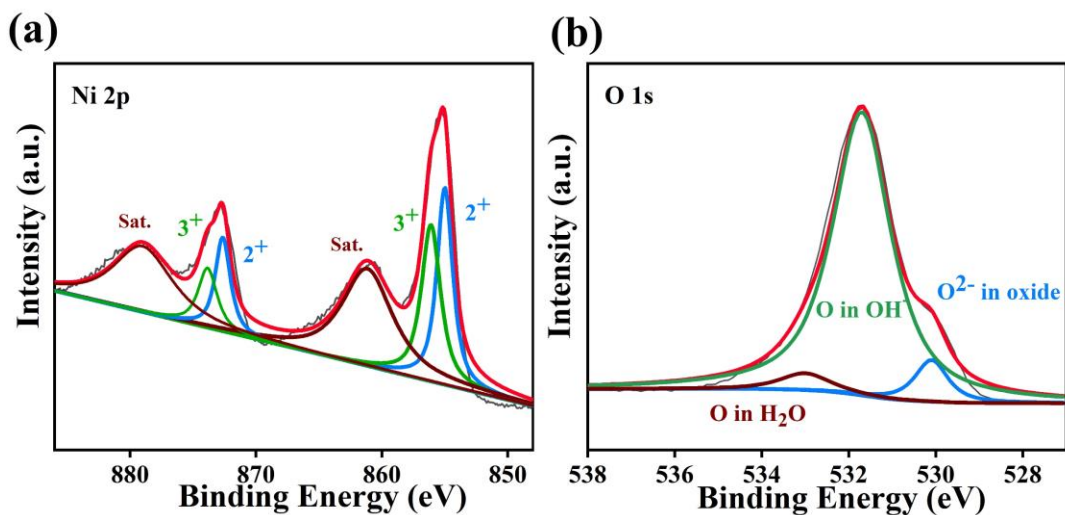


Fig. S4 XPS spectra of NiO<sub>x</sub> catalyst: (a) Ni 2p and (b) O 1s spectra.

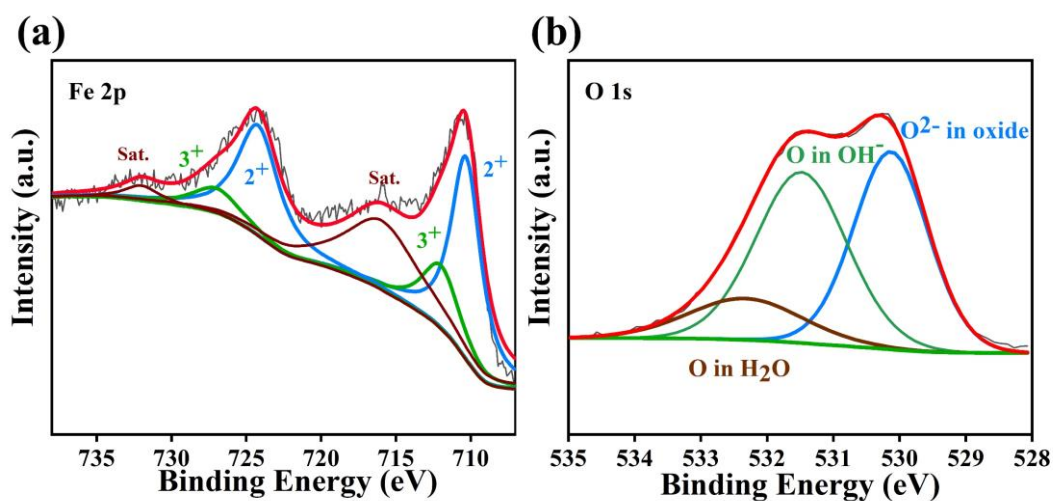


Fig. S5 XPS spectra of FeO<sub>x</sub> catalyst: (a) Fe 2p and (b) O 1s spectra.

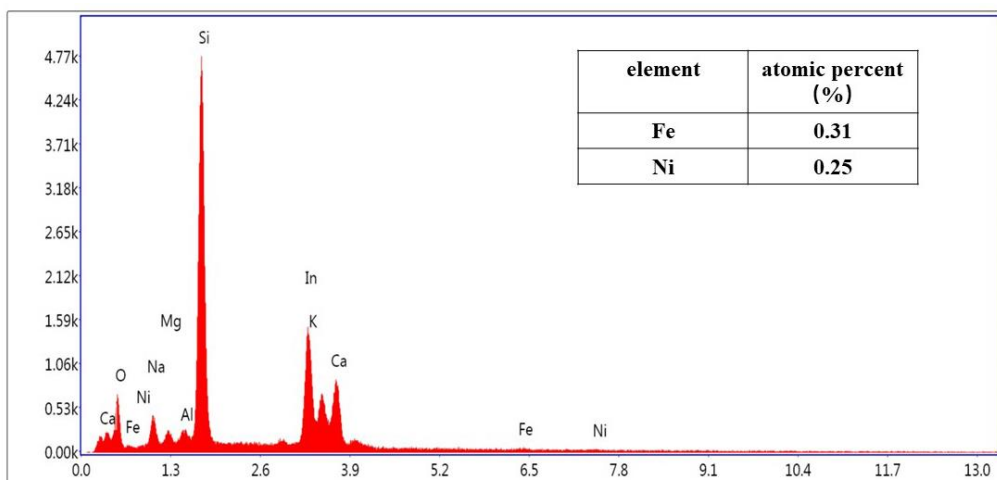
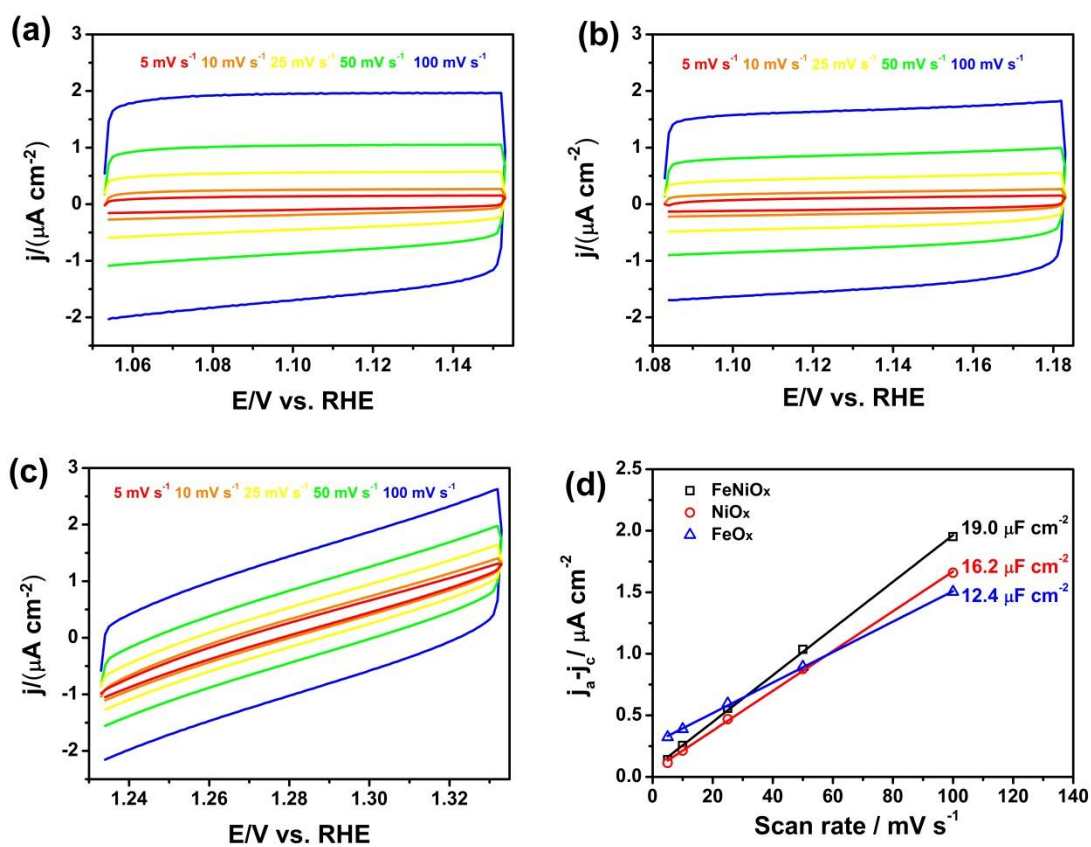
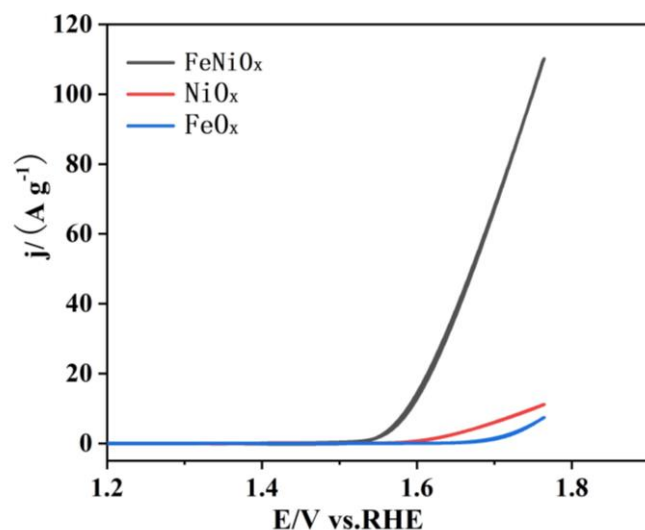


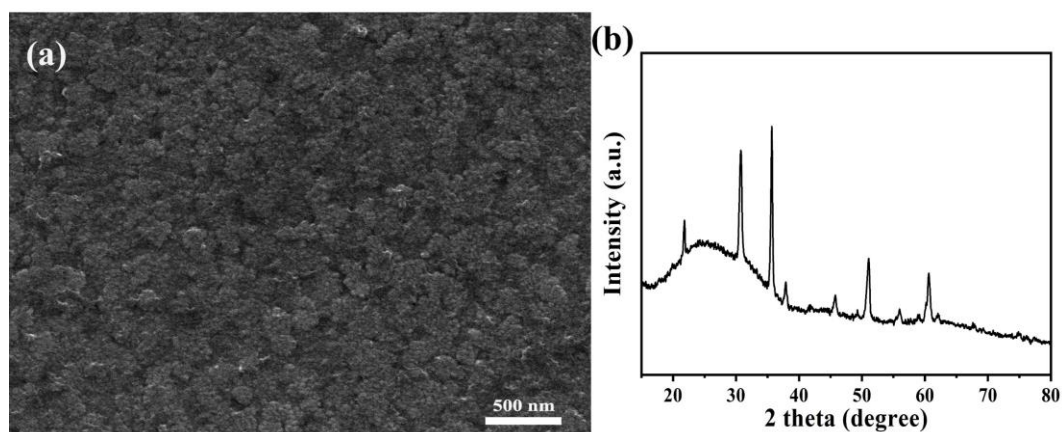
Fig. S6 EDS of FeNiO<sub>x</sub> catalyst



**Fig. S7** (a) The CV of FeNiO<sub>x</sub> catalyst measured in a non-Faradaic potential window of 1.05 to 1.15 V vs. RHE. (b) The CV of NiO<sub>x</sub> catalyst measured in a non-Faradaic potential window of 1.08 to 1.18 V vs. RHE. (c) The CV of FeO<sub>x</sub> catalyst measured in a non-Faradaic potential window of 1.23 to 1.33 V vs. RHE. All the measurements were performed in 1 M KOH at the following scan rates: 5 (red), 10 (orange), 25 (yellow), 50 (green), 100  $\text{mV s}^{-1}$  (blue). (d) Charging current density difference ( $j_a - j_c$ ) plotted against scan rate. The linear slope is one times that of the  $C_{dl}$ .  $C_{dl}$  for FeNiO<sub>x</sub>, NiO<sub>x</sub>, and FeO<sub>x</sub> are 19.0, 16.2, 12.4  $\mu\text{F cm}^{-2}$ , respectively. ECSA for FeNiO<sub>x</sub>, NiO<sub>x</sub>, and FeO<sub>x</sub> are 0.475, 0.405, 0.31  $\text{cm}^2$ , respectively.



**Fig. S8** The mass activity of FeNiO<sub>x</sub>, NiO<sub>x</sub> and FeO<sub>x</sub> in 0.1 M KOH.



**Fig. S9** SEM (a) and XRD pattern (b) of the FeNiO<sub>x</sub> after electrolysis.

Table S1 Concentrations of precursors in electrodeposition baths.

Catalysts	NiSO <sub>4</sub>	Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	NaOAc
FeNiO <sub>x</sub>	10 mM	5 mM	0.1 M
NiO <sub>x</sub>	10 mM		0.1 M
FeO <sub>x</sub>		5 mM	0.1 M

## References

1. Gao M R, Cao X, Gao Q, et al. ACS nano, 2014, 8(4): 3970-3978.