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Rapid Synthesis of Co_3O_4 Nanosheets Arrays on Ni Foam by in Situ Electrochemical Oxidizing the Air-Plasma Engraved $Co(OH)_2$ for Efficient Oxygen Evolution

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Experimental

Chemicals and Reagents. Cobalt nitrate hexahydrate ($Co(NO_3)_2 \cdot 6H_2O$) was purchased from Beijing Chemical Reagent (Beijing, China). Nickel foam (NF) was obtained from Shenzhen Green and Creative Environmental Science and Technology Co., Ltd. Unless stated, all the solvents and chemical materials are used without further purification. The aqueous solutions of 1.0 M KOH (pH = 14) were prepared with Millipore Milli-Q (18.2 MΩ cm) deionized water.

Synthesis of $Co(OH)_2$@NF. To remove the impurities and oxide, the NF was immersed into 3 M HCl solution 6 h before electrodeposition. Then, the NF was cleaned sequentially in ultrasonic baths with deionized water and ethanol for 30 min each, baked at 60 ^oC for 8h. The following electrodeposition of $Co(OH)_2$ nanosheets onto NF substrates was performed in the three-electrode system, with NF, a graphite rod and Ag/AgCl as working electrode, counter electrode, and reference electrode, respectively. An operation potential of -1.1 V was applied in electrodeposition, which was performed in 6 mmol $Co(NO_3)_2 \cdot 6H_2O$ for 30 min. After electrodeposition, the sample was taken out and washed with deionized water and then dried at 60 ^oC for 6 h. By comparing the quality of $Co(OH)_2$@NF with the original NF and doing the ICP-AES measurement, the mass of $Co(OH)_2$ deposited onto each NF was around 10 mg/cm^2. To improve the catalytic activity of $Co(OH)_2$@NF, the air plasma treatments with different time (0 min, 5 min, 10 min, 15 min and 20 min) were conducted at 40 W.

Electrochemical measurement. In the electrochemical experiments, the catalytic properties of samples were investigated with a CH Instruments 800 voltammetric analyzer (Shanghai, China) in three-electrode system with a graphite rod, an Ag/AgCl (saturated KCl) electrode and modified NF as the counter electrode, reference electrode and working electrode, respectively. Thus, the OER activity and stability of the samples were studied with linear sweep voltammetry (LSV), cyclic voltammetry (CV) and amperometric i−t curve in 1.0 M KOH. In this work, all the potentials were obtained with iR compensation. In order to ensure the equilibrium of O_2/H_2O at 1.23 vs. RHE, the oxygen flow was maintained at a small flow rate during the whole testing process.

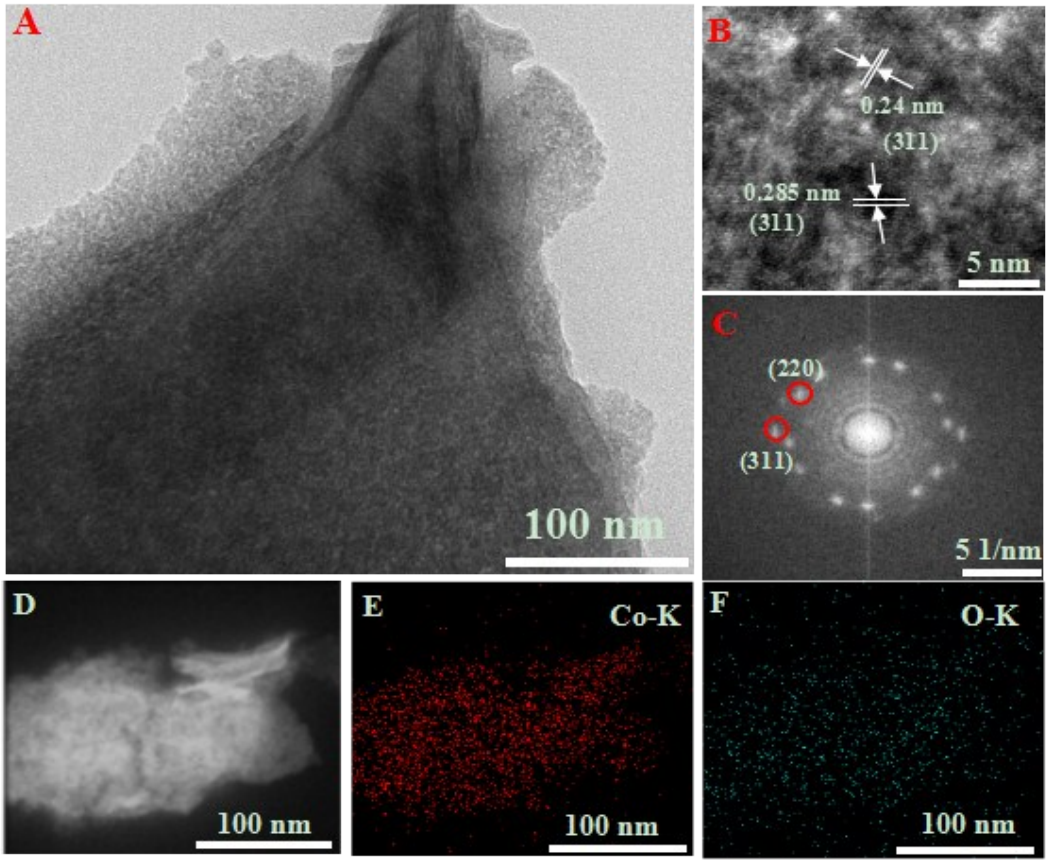

Figure S1. (A) high-magnification TEM image of Co_3O_4@NF; (B) HRTEM image of the Co_3O_4@NF; (C) the corresponding Fast Fourier Transform (FFT) analysis of the (311) plane and (220) plane of Co_3O_4. (D-F) HAADF-STEM and the corresponding elemental mapping images of Co_3O_4@NF.

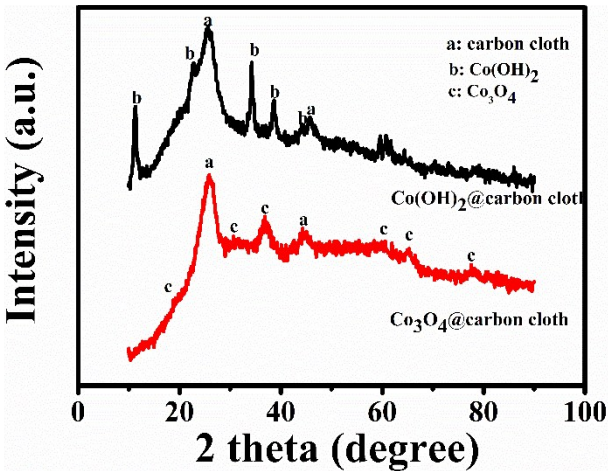

Figure S2. The XRD of $Co(OH)_2$@carbon cloth and Co_3O_4@carbon cloth.

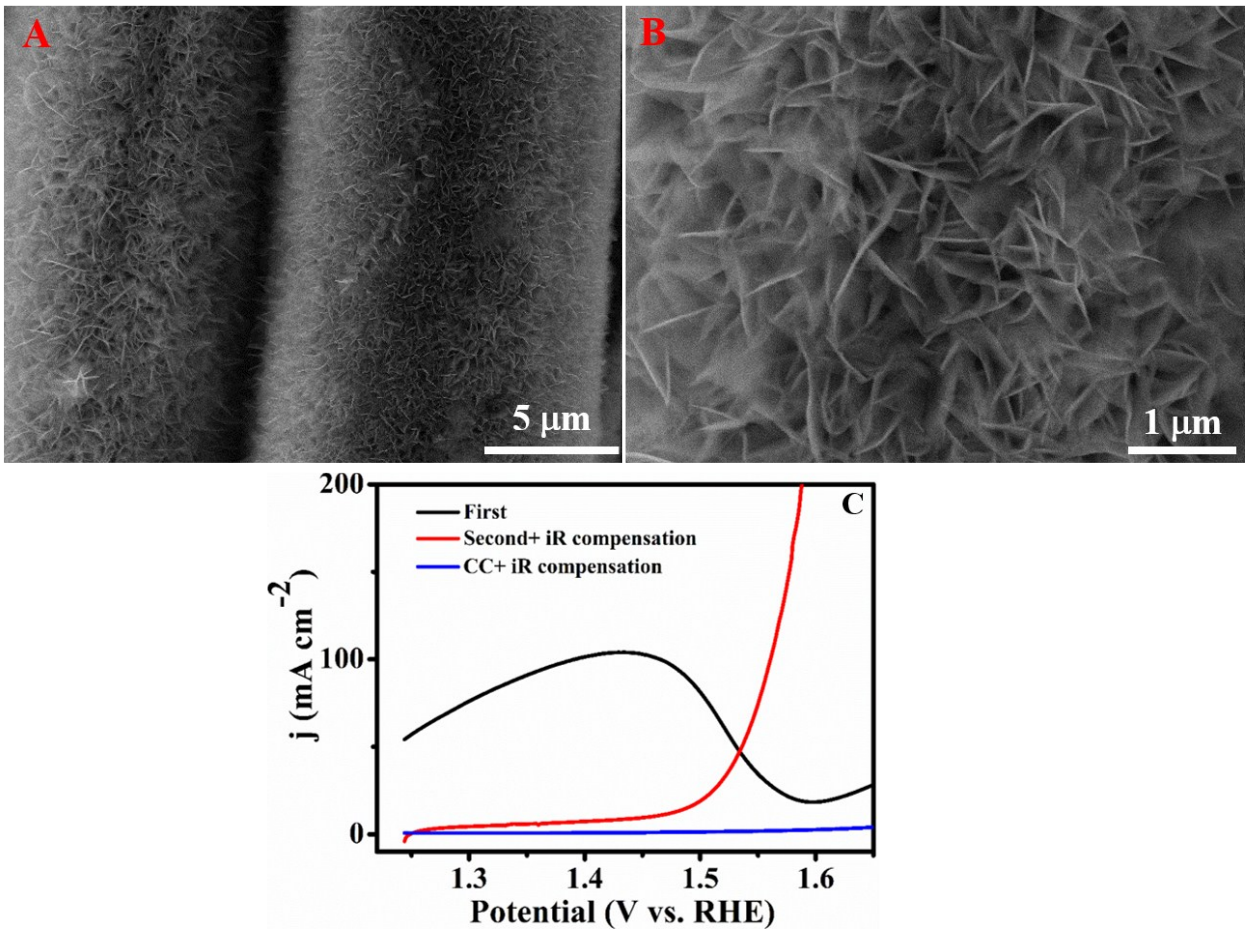

Figure S3. (A, B) The SEM images of the $Co(OH)_2$@carbon cloth. (C) LSV polarization curves of the pristine carbon cloth (CC) and $Co(OH)_2$@CC for the first and second scanning.

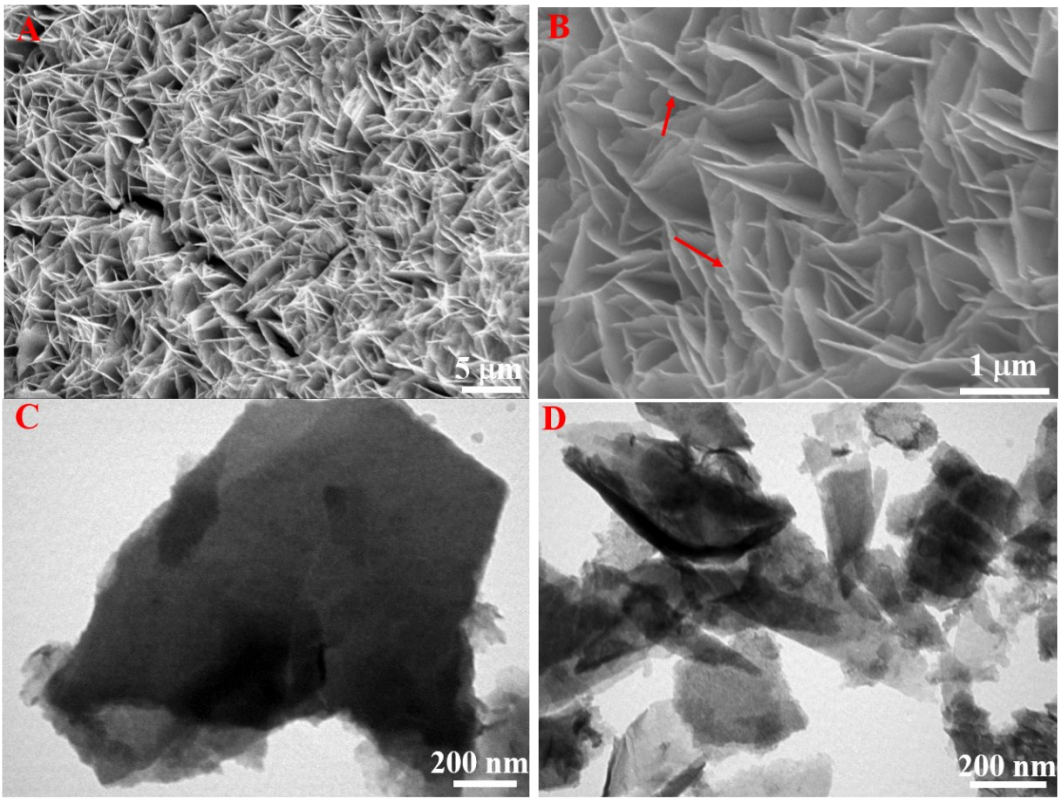

Figure S4. The SEM images of $Co(OH)_2$@NF without (A) and with (B) the engraving by air plasma. The TEM images of $Co(OH)_2$@NF without (C) and with (D) the engraving by air plasma.

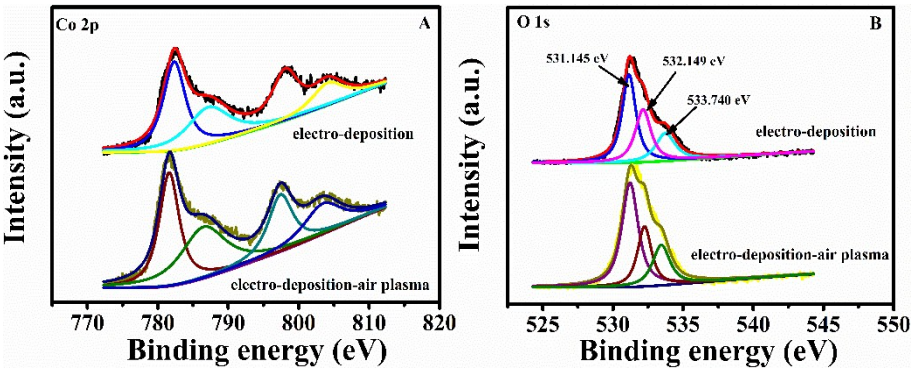

Figure S5. The XPS survey spectrums of $Co(OH)_2$@NF without and with the engraving by air plasma.

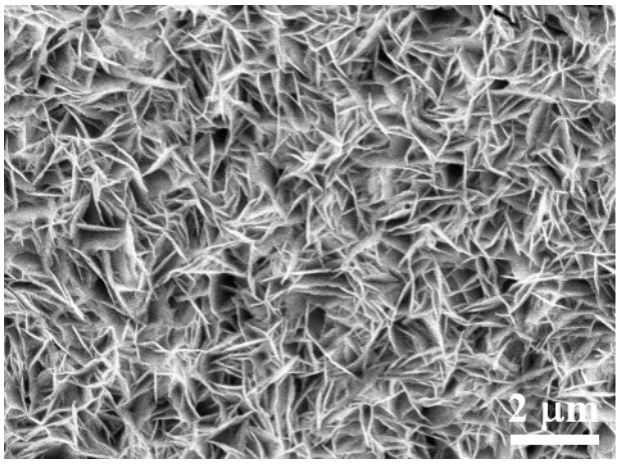

Figure S6. The SEM image of Co_3O_4@NF-15 after stability test.

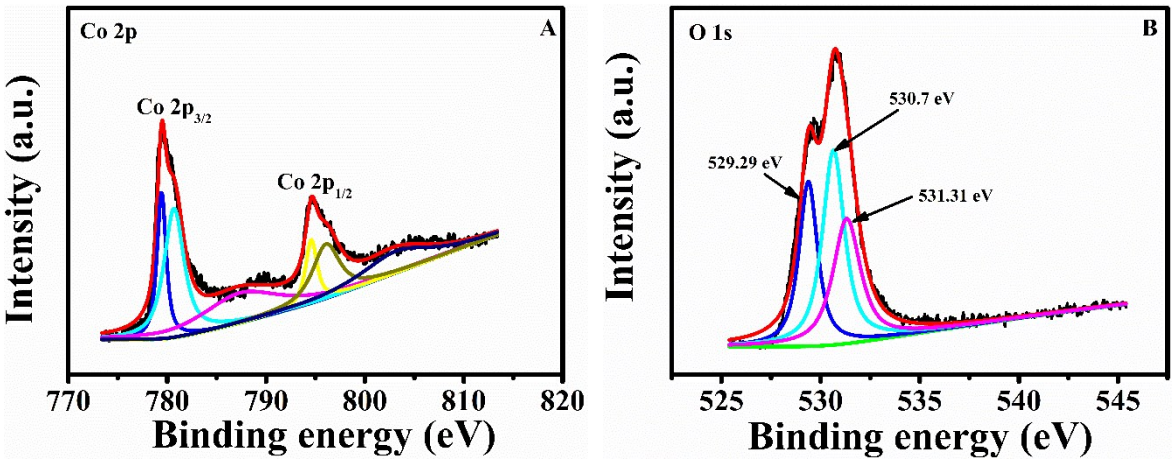

Figure S7. The XPS spectra of Co_3O_4@NF-15 after stability test in Co 2p (A) and O 1s (B) region.