## Electronic Supplementary Information

## Facile Synthesis of Mechanically Robust and Highly Porous NiO Film with Excellent Electrocatalytic Activity towards Methanol Oxidation

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Fig. S1 Linear polarization (LP) curves of nickel in the solutions with 80 wt.%  $H_3PO_4$  and different  $NH_4F$  concentration.



Fig. S2 The output current of nickel anodized in 0.5 wt.% NH4F + 85 wt.%  $H_3PO_4$ . The anodization potential was swept from open circuit potential to 3.5 V at 10 mV s<sup>-1</sup>, and then kept constant at 3.5 V for 5 min.



Fig. S3 XPS Ni 2p spectra of the sample after annealing.

Two Ni  $2p_{3/2}$  peaks were observed at 855.9 and 857.7eV, corresponding to those of Ni(OH)<sub>2</sub> and NiF<sub>2</sub>,<sup>1</sup> respectively.



Fig. S4 Cycling test of anodic NiO in 0.1 M NaOH + 1 M CH<sub>3</sub>OH at 50 mV s<sup>-1</sup>



Fig. S5 SEM image of anodic NiO before the cycling test.



Fig. S6 (a) SEM image and (b) Cyclic voltammetry curve of anodic NiO film produced conformally on the nickel foam. Electrolyte: 0.1 M NaOH with 1 M  $CH_3OH$ , scan rate: 50 mV s<sup>-1</sup>.

 M. C. Biesinger, L. W. M. Lau, A. R. Gerson and R. S. C. Smart, *Phys. Chem. Chem. Phys.*, 2012, 14, 2434-2442.