Supporting Information for

NiO Nanocone Array Electrode with High Capacity and Rate Capability for Li-Ion Batteries

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Experiments details

Fabrication procedure: NiO nanocone array (NCA) electrodes for Li-ion batteries were fabricated by eletrodeposition of Ni nanocone array on Ni foam (100 PPI pore size, 380 g m⁻² surface density, and 1.5 mm thick) followed by thermal oxidation in air. The electrodeposition of Ni NCAs was carried out by a previously reported method with slight modifications.¹⁻³ In a typical procedure, Ni foams were first sonicated in high-purity ethanol for 30 min. Ni NCAs were then electrodeposited on the pretreated Ni foams at a constant deposition current density of 0.06 A cm⁻². The electrodeposition solution was composed of analytical pure NiCl₂•6H₂O (1 mol L⁻¹ for providing Ni ion), H₃BO₃ (0.5 mol L⁻¹ as pH buffer), and ethylenediamine dihydrochloride (1.5 mol L⁻¹ as crystal modifier) dissolved in doubly deionized water. The temperature of the solution was maintained at 60 °C. The electrodeposited Ni NCA on Ni foams were rinsed with doubly deionized water and dried in a vacuum oven at 60 °C for 3 h, then heated to 400 °C at a rate of 5 °C min⁻¹ and held for 1 h in a horizontal tube furnace. The mass of the Ni NCA on Ni foams before and after oxidation was weighed by a microbalance (Mettler, XS105DU) with an accuracy of 0.01 mg. According to the reaction of $2Ni + O_2 = 2NiO$, the active weights of NiO (m_{NiO}) are derived from $m_{NiO} = \Delta m \times 74.69/16$, where Δm is the weight difference of the Ni NCAs on Ni foams before and after oxidation reaction.

Structural Characterization: The structure and morphology of the NiO NCA electrodes were characterized by X-ray powder diffraction (Rigaku D/Max-2400 with Cu Kα radiation) and field-emission scanning electron microscopy (Hitachi, FE-SEM S-4800).

Electrochemical Characterization: Electrochemical characterizations were carried out using CR-2032-type coin cells, which were assembled in a high-purity argon filled glove box $(H_2O < 0.5 \text{ ppm}, O_2 < 0.5 \text{ ppm}, MBraun, Unilab)$ by using the NiO NCA electrodes as the working electrode and lithium foil as the counter and reference electrode. Celgard 2320 was used as the separator membrane. The electrolyte was 1 M lithium hexafluorophosphate (LiPF₆) dissolved in ethylene carbonate : dimethyl carbonate : ethyl methyl carbonate in a 1:1:1 volume ratio. The galvanostatic charge-discharge cycling and cyclic voltammetry were carried out at room temperature by using a multichannel battery tester (Neware, BTS-610) and an electrochemical workstation (CHI, 660C), respectively.

Reference

- 1 T. Hang, M. Li, Q. Fei and D. Mao, *Nanotechnology*, 2008, **19**, 035201.
- 2 S. Zhang, Z. Du, R. Lin, T. Jiang, G. Liu, X. Wu and D. Weng, *Adv Mater*, 2010, **22**, 5378-5382
- 3 D. Wang, Z. Yang, F. Li, D. Liu, X. Wang, H. Yan and D. He, Mater. Lett., 2011, 65, 1542-1544

Supplementary figures



Figure S1. Cross-sectional SEM image of the as-prepared electrodes. Obvious boundary can

be seen between NiO and the electrodeposited Ni.



Figure S2. X-ray diffraction patterns of the electrodeposited Ni NCA on Ni foams before and after oxidation. Two typical diffraction peaks located at 44.4 ° and 51.7 ° can be observed before oxidation, corresponding to Ni (111) and (200) faces, respectively (JCPDS 4-850). After thermal oxidation, three new diffraction peaks appear at 37.1 °, 43.2 ° and 62.7 °, which can be indexed to the (101), (012) and (110) faces of the cubic NiO phase, respectively (JCPDS 44-1159).



Figure S3. Capacity retention and coulombic efficiency of the NiO NCA electrode at a rate as high as 10 C after the first five cycles at a low rate of 0.4 C. The electrode can deliver a capacity of about 630 mAh g⁻¹ at the rate of 10 C and retain a capacity of 547 mAh g⁻¹ after 50 cycles.



Figure S4. Capacity retention of the NiO NCA electrode after cycling at high rates shown in Fig. 3b. The NiO NCA electrode can yet reach a high reversible capacity of about 1198 mAh g^{-1} after 50 cycles at a rate of 0.4 C.