



Short communication

Batch fabrication of mesoporous boron-doped nickel oxide nanoflowers for electrochemical capacitors



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ARTICLE INFO

Article history:

Received 5 June 2014

Received in revised form 14 July 2014

Accepted 31 July 2014

Available online 2 August 2014

Keywords:

Nanoparticles
Nanocomposites
Porous materials
Thin films

ABSTRACT

Boron-doped nickel oxide (B-NiO) nanoflowers are prepared by simple thermal decomposition of nickel hydroxide. B-NiO is porous sphere with a diameter of about 400 nm. B-NiO nanoflowers are composed of approximately 30 nm nanoplates and the thickness of the nanosheets is approximately 3 nm. The specific surface area of the material is as high as $200 \text{ m}^2 \text{ g}^{-1}$ and the pore size distribution curves of B-NiO has three typical peaks in the range of mesoporous (5 nm, 13 nm and 18 nm). As an electrode for supercapacitors, the crystalline B-NiO nanoflowers have favorable characteristics, for instance, a specific capacitance of 1309 F g^{-1} at a current density of 3 A g^{-1} and no significant reduction in Coulombic efficiency after 2500 cycles at 37.5 A g^{-1} . This remarkable electrochemical performance will make B-NiO nanoflowers a promising electrode material for high performance supercapacitors.

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1. Introduction

Electrochemical capacitors (ECs) and batteries are increasingly used in hybrid electric vehicles, power quality devices, and portable electronics [1–3]. The development of high-energy electrode material density is urgent. There are two types of ECs, one is electrochemical double-layer capacitors (EDLCs) and the other is pseudocapacitors [4]. As to the pseudocapacitors, especially redox-type capacitors, the electrode materials are most the transition-metal oxides and electrically conducting polymers. The pseudocapacitors have higher energy densities and specific capacitances than EDLCs [5]. Because of the high theoretical specific capacitance and cycling stability, transition metal oxide has been considered as a potential kind of candidate. For instance, RuO_2 is a promising active material with specific capacitance as high as 760 F g^{-1} in an aqueous acid electrolyte. However, its high cost and toxicity besides the unsatisfactory specific capacitance limit its applications [4]. Thus, the cheap transition-metal oxides have been widely investigated, such as MnO_2 , Co_2O_3 [6,7] and NiO materials [8–11]. Among these materials, NiO is optimal electrode material owing to its low cost, lower toxicity, easy synthesis, and high theoretical specific capacity (2573 F g^{-1}). Among the

numerous studies about NiO that have been published, the specific capacitance is in a wide range [11–14]. Because of the low electrical conductivity and the slow Faradaic reactions on the electrode surface, NiO often has poor rate capability. The performance of NiO can be improved by providing a large surface area and further increasing the electrical conductivity [5,15]. The nanostructured materials always have high-specific surface area for facilitating the electrochemical redox reactions and mitigate diffusion resistance by shortening the diffusion path [16]. The doping modification is another an important way to improve its performance.

Herein, we report a simple method for synthesizing boron-doped mesoporous nickel oxide (B-NiO) through coprecipitation and thermal decomposition of nickel hydroxide. This kind of B-NiO exhibits excellent capacitive behavior (1309 F g^{-1} at a current density of 3 A g^{-1}) and stable cycle life (no significant reduction in Coulombic efficiency after 2500 cycles at 37.5 A g^{-1}).

2. Experimental

2.1. Reagents

The surfactant copolymer poly (ethyleneoxide)-poly (propyleneoxide)-poly (ethylene oxide), commercially known as Pluronic ($\text{P}_{123} \text{ EO}_{20} \text{ PO}_{70} \text{ EO}_{20}$). Polytetrafluoroethylene (PTFE, 60 wt. % water suspension) was from Aladdin. Other chemicals were all analytical reagents from Beijing Chemical Company. All stock solutions were prepared with deionized water resistivity not less than $18.2 \text{ M}\Omega \text{ cm}$.

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2.2. Synthesis of materials

We got boron-doped NiO nanoflowers by calcination the Ni(OH)₂ at 873 K for 4 h with the heating rate 10 K min⁻¹ in air. Ni(OH)₂ was based on a self-assembly between a triblock copolymer template P₁₂₃ and two precursors (sodium borohydride and nickel species) in a flask [17]. Briefly, Ni(NO₃)₂·6H₂O (8.6 g) solution was put into the 440 ml solution of P₁₂₃ (4 g), and then stirring at 313 K for more than 3 h. Then dropped sodium borohydride (1.5 g) solution into the mixture. After 3 h, the same quantity of sodium borohydride solution was added into the mixture, too. After 12 h, light green sediments were formed. The mixture was filtered and washed with water and ethanol alternately until no foam of surfactant P₁₂₃ in the filtrate, then we got Ni(OH)₂ with boron. We could obtain the Ni(OH)₂ without boron as control material by replacing sodium borohydride with sodium hydroxide.

2.3. Preparation of nickel electrodes and electrochemical measurements

A three electrode system was used. Ag/AgCl electrode filled with saturated potassium chloride was used as reference electrode and platinum foil was used as counter electrode with 1 M aqueous solution of KOH was used as electrolyte.

The cyclic voltammograms (CVs) were recorded on a CHI660D electrochemical workstation (CHI, Shanghai) at 298 K.

$$C = \frac{\int Idt}{m\Delta V}$$

The above formula was employed as equation for calculation average specific capacitance values from the CV curves. Where *I* means the oxidation or reduction current, *m* indicates the mass of the active electrode material, *dt* indicates time differential, and ΔV is the voltage range of one sweep segment. And there is another

method for calculation the specific capacitance values from the galvanostatic charge and discharge curves as the following formula:

$$C = \frac{I\Delta t}{m\Delta V}$$

while, where *I* represents charge or discharge current, Δt indicates the time for a full charge or discharge, ΔV is the voltage change after a full charge or discharge and *m* is the mass of the active material.

3. Results and discussion

As shown in Fig. 1(A), all of the diffraction peaks of B-NiO could be perfectly assigned to the JCPDS file(65–2901) of the cubic phase. No obvious diffraction peaks of other phases were observed, indicating the high quality of the nanoflowers. The B-NiO nanoflowers was examined using XPS for estimating the various chemical states of bonded elements as shown in Fig. 1(B). There was an obvious peak at 191.8 eV corresponding to B 1s. The XPS spectra of Ni 2p was shown in Fig. 1(B, inset). The Ni 2p_{3/2} peak was assigned to Ni(II) ions in the B-NiO samples and the position of the peak was at 854.3 eV. The peak at 855.8 eV was attributed to Ni³⁺ species on the B-NiO surface [18]. In the corresponding O 1s spectra shown in Fig. 1(B, inset), peaks correspondence were observed. There were a distinct peak at 529.6 eV and a shoulder peak at 531.3 eV. Here, we could attribute the shoulder peak to the existence of Ni₂O₃ and defect sites on the B-NiO surface [18].

Fig. 1(C and D) showed the SEM micrographs of the corresponding B-NiO nanoflowers samples. The average particle size of the B-NiO spheres was 400 nm. As Fig. 2(A and B) presented, TEM photo of B-NiO, the sheet structure was observed clearly. The HRTEM images of B-NiO presented in (Fig. 2(C and D)), they showed that the edges of the nanosheets were mainly exposed

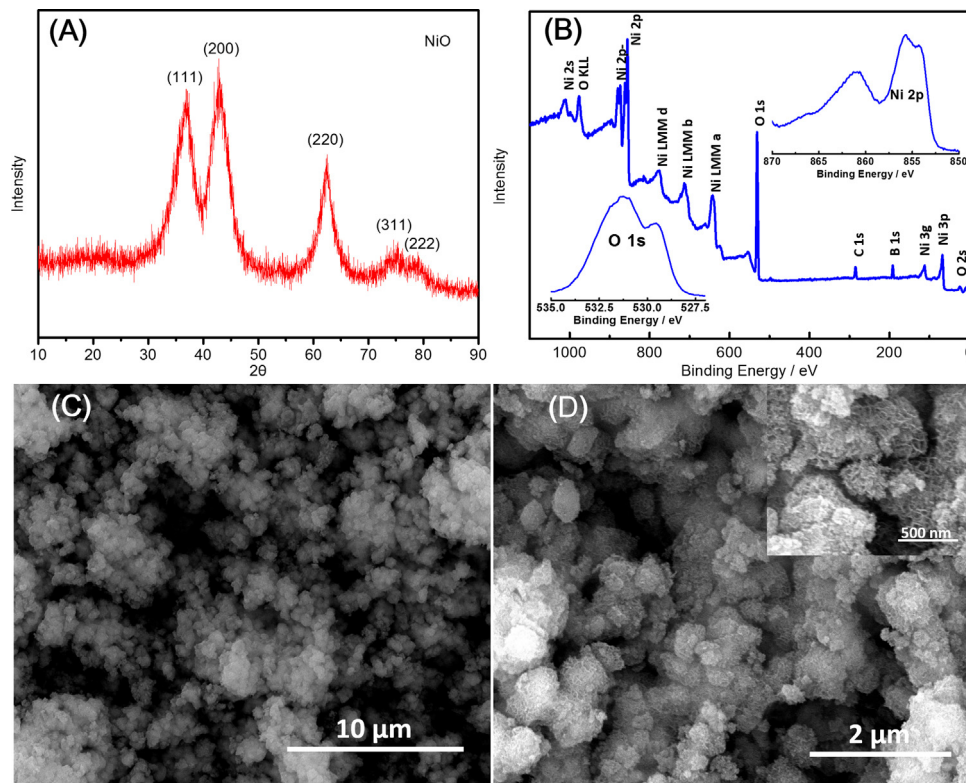


Fig. 1. XRD patterns of B-NiO (A) XPS spectroscopy (B) XPS Ni 2p (inset) and XPS O 1s (inset) of B-NiO. SEM images of the B-NiO (C, D).

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