



Large-scale synthesis of highly crystalline rectangular prism-like CeO₂ microrods with excellent lithium storage behavior



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ABSTRACT

In this paper, highly crystalline rectangular prism-like CeO₂ microrods were synthesized by a microwave assisted solution without any surfactants or templates. The reaction time was only 30 min. The structure and morphology were characterized by XRD, SEM, TEM and XPS. The calculated lattice parameter was $a = 0.5405$ nm. The microrods growth was in the [111] direction. A possible formation mechanism was suggested. As an anode material for Li-ion batteries, lithium storage behaviors of the as-prepared CeO₂ were evaluated in 0.01–1.5 V by charge-discharge test, CV and EIS. The discharge capacity retention was 98.97% with reference to the 10th cycle (319.5 mAh g⁻¹) after 100 cycles at 0.2 mA cm⁻². The excellent lithium storage behavior should be attributed to 1-dimensional (1D) microrod structures, the path of lithium ions diffusion and the maintain of the microrod morphology during lithiation/delithiation. Furthermore, the obtained rectangular prism-like CeO₂ microrods can also be extended to other application fields.

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1. Introduction

Considering the threat arising from limited oil storage and environmental pollution, developing lithium power batteries (LPBs) is becoming more urgent. One of research issues in LPBs is to explore new materials with large capacity density and long cycling stability to substitute commercial carbon anode [1]. Recently, CeO₂ has also showed a promising application prospect for lithium storage material due to higher theory capacity and the quick mutation between Ce³⁺ and Ce⁴⁺ [2–5]. In generally the properties and applications of materials strongly depend on their sizes and morphologies. For example, Brick-like CeO₂ delivered an electrochemical capacity of 460 mAh g⁻¹, which was superior to that of plate-like particles [3]. It demonstrated that the morphology control of micro/nano materials would effectively influence its electrochemical performances. Up to now, CeO₂ with various morphologies have been prepared [6,7]. Including as zero, one, two and three-dimensional materials [6]. Of these, 1D CeO₂ has attracted great interests because of a peculiar geometry structure, such as low dimensionality, novel size effects and enhanced

kinetics, resulting into enormous potential as components of nanodevices and sensors. It was worth noting that 1D prism α -Fe₂O₃ delivered excellent lithium storage performances for providing a short path for lithium-ion diffusion [8], which is worthy of reference for CeO₂. However, to the best of our knowledge, there has not been any report on lithium storage evaluation of 1D prism CeO₂.

Various synthetic routes have been developed to prepare CeO₂, including hydrothermal, microwave assisted thermal decomposition, a one-pot approach electrochemical method, combustion synthesis, sol-gel, homogeneous precipitation [9]. Among these methods, microwave-assisted synthesis is a very economical and clean route for significantly decreased reaction time. Additionally, the most important point to be noted is that, compared with the conventional heat method, the application of microwaves offers a great possibility for large-scale synthesis [10]. Nevertheless, only a few literature reported the fabrication of CeO₂ via microwave method. For example, Goharshadi et al. synthesized CeO₂ nanoparticles by a microwave-assisted heating technique in NaOH aqueous solution [11]. In particular, Riccardi and coworkers prepared CeO₂ rhombus-shape by a simple microwave-hydrothermal method in urea water solution using cetyltrimethylammonium bromide as surfactant [9]. However, it is difficult for large scale production because the removal of the surfactant is still a challenge,

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so it is highly needed to develop a surfactant-free and facile way to synthesize well-crystalline CeO₂.

In this study, uniform highly crystalline rectangular prism-like CeO₂ microrods have been successfully synthesized by a microwave assisted solution. The whole procedure was easily reproducible and free of surfactants and templates. The reaction time was only 30 min. The yield was very high. This method was likely to be easy to scale up for industrial production. Furthermore, lithium storage capabilities of the as-prepared CeO₂ were investigated, which indicated the excellent performance and hence hold potential application.

2. Experimental

All reagents were analytical grade and were used without further purification before the synthesized process. In a typical synthesis, 500 mL distilled water and 50 mL alcohol were firstly mixed in a 1 L Schott Duran glass bottle, and then 2.1732 g Ce(N-O₃)₃·7H₂O, 1.5039 g urea were dissolved into the above mixed solution under stirring to form a homogeneous solution. The glass bottle was sealed with a polypropylene screw cap and placed into in a domestic microwave oven (Galanz WD900SL23-2, China). The system was treated for 30 min at 720 W (a longer time heat at higher power was prohibited and solution was limited in 0.67 L), then the bottle was cooled naturally to room temperature, and the white precipitate was centrifuged and washed with distilled water and alcohol three times each. Finally, the obtained precipitate was dried at 100 °C overnight and then heated at 500 °C for 8 h to form the final powder. A yield was found to be 82–94%.

XRD analysis using Cu K_α radiation ($\lambda = 1.54056 \text{ \AA}$) was employed to identify the crystalline phases of the prepared powder with a Bruker D8-advance (Germany) at room temperature in the range of $20^\circ \leq 2\theta \leq 80^\circ$. The morphology was investigated with SEM (Hitachi SU8010, Japan) and TEM (FEI, TECNAI G220 S-Twin, The U. S. A). The compositions of the obtained powders were measured by an X-ray photoelectron spectrometer (XPS, Thermo VG Multi Lab 2000, The U. S. A). The two-electrode button cells were assembled by using lithium foil as cathode in an argon-filled glove box, the as-prepared CeO₂ mixed with 12% acetylene black and 8% polytetrafluoroethylene as the anode, 1 M LiPF₆ in a 1:1 (v/v) mixture of ethylene carbonate and dimethylcarbonate as the electrolyte, Celgard 2300 membrane as the cell separator. The charge–discharge cycles were performed at various current densities in a potential range of 0.01–1.5 V. All the electrical measurements were carried out by a battery testing system (BTS-5 V/10 mA, Neware technology limited corporation, China) at room temperature. Electrochemical impedance spectroscopy (EIS) analysis was performed on the electrodes using Autolab PGSTAT302 electrochemistry workstation. The Nyquist plots were obtained at amplitude of 10 mV, and the frequency range applied was from 0.01 to 100 kHz.

3. Results and discussion

The crystalline structures of the powder calcinated at 500 °C were examined by XRD. As shown in Fig. 1, all the peaks, at $2\theta = 28.45, 32.97, 45.36, 56.44, 59.04, 69.46, 76.99,$ and 79.32 , were in good agreement with CeO₂ standard cards (PDF#34-0394, blue lines). No other diffractions delegating byproducts were found, confirming the high purity of CeO₂. After refinement with face-centered cubic fluorite structure (space group Fm-3m) by TOPAS 3 software, the calculated lattice parameter was $a = 0.5405 \text{ nm}$ with a convincing Rwp value (6.284%). The value was lower than that (0.5412 nm) of CeO₂ prepared by similar wave method [12].

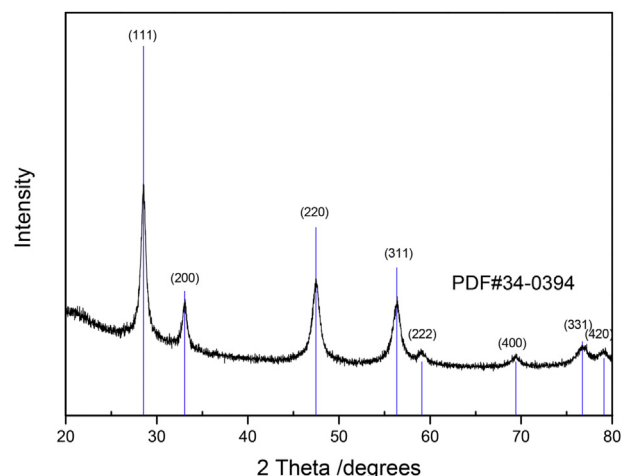


Fig. 1. XRD pattern of the calcined powder at 500 °C (with the reference data PDF card # 43-1002 for CeO₂ on bottom).

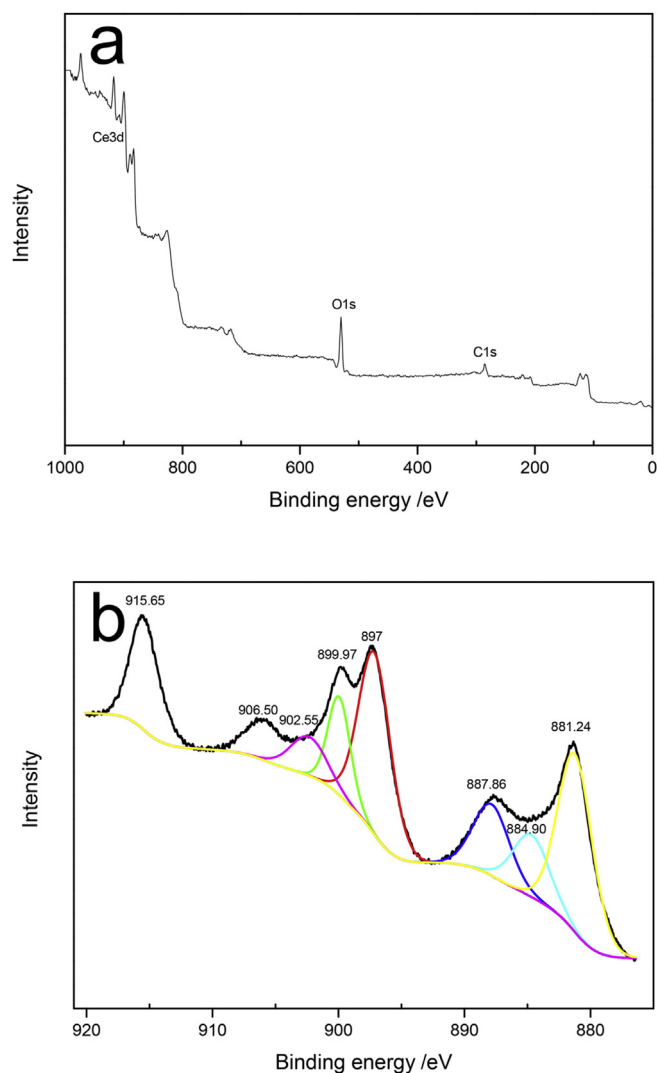


Fig. 2. (a) XPS survey spectrum and (c) Ce 3d core level XPS spectrum of the as-fabricated powder.

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