



α -Fe₂O₃@C nanorings as anode materials for high performance lithium ion batteries



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ABSTRACT

α -Fe₂O₃@C core–shell nanorings are prepared by a facile large-scale two-step route incorporating a hydrothermal method and a carbon coated progress. Its structure and morphology are characterized by X-ray diffraction, scanning electron microscopy, transmission electron microscope, and thermogravimetry. It is found that the as-prepared composite is composed of α -Fe₂O₃@C nanorings of about 148 nm in outer diameter, 50 nm in thickness, and 115 nm in length. These α -Fe₂O₃@C nanorings are wrapped with ~3 nm thick carbon shell. And the electrodes exhibit longer cycle life (815 mAhg⁻¹ after cycling 160 times) at high current rate (1000 mA g⁻¹) compared with that of bare α -Fe₂O₃ nanorings (810 mAhg⁻¹ after cycling 30 times). The improved performance of the composite is attributed to the bondage on carbon shell, which can enhance the electronic conductivity and structural stability of α -Fe₂O₃ nanorings.

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1. Introduction

Recently, transition metal oxides (TMOs) as potential anode materials for lithium ion batteries (LIBs) have attracted enormous research interests, due to their high theoretical capacity [1–4]. Among them, α -Fe₂O₃ has long been regarded as a promising anode material for LIBs due to its low conversion potential, low voltage hysteresis (<0.8 V), high theoretical capacity (1007 mAhg⁻¹), natural abundance, environmentally benign and low electrochemical motivation force (1.032 V vs Li/Li⁺) [5]. However, like other TMOs, α -Fe₂O₃ suffers from poor rate capability because of the intrinsic low conductivity, especially cycling at high current densities. The α -Fe₂O₃ also has poor cycle life which is caused by the large volume change during discharge/charge process [6–8].

It is well known that coating α -Fe₂O₃ anode materials with carbon shell is an advanced method to enhance the capacity retention, rate capability and the cyclability. This is because coating carbon on the surface of the anode can significantly improve the conductivity of α -Fe₂O₃. Besides, the elastic feature of carbon shell can effectively address the strain arising from the large volumetric

change during lithium insertion/de-insertion process, partly improving the cyclability of α -Fe₂O₃ [9]. On the other hand, nano-sized materials, compared with bulk materials, can possess distinct rate capability and exhibit enhanced lithium storage performance, due to their large surface-to-volume ratio and nanoscale dimensions [10]. To improve the cyclability and rate capability of α -Fe₂O₃, various types of α -Fe₂O₃ nanostructures have been investigated as anode materials, such as thin films [11], bubble-nanorod composite [12], hollow nanococoons [13], microspheres [14], nanorods [15], and nanotubes [16].

The ring-like nanostructures are attracting fast growing research interests, due to their unique geometry and structural flexibility resulting from strain-free mechanical properties [17,18]. Inspired by the advantages of ring-type nanostructure and carbon coating, we successfully synthesized α -Fe₂O₃@C core–shell nanorings via a facile large-scale process. It is intriguing to find that the specific structure of α -Fe₂O₃@C can improve the electronic conductivity and reduce the dissolution and aggregation of α -Fe₂O₃ during electrochemical process. As a consequence, α -Fe₂O₃@C nanorings exhibit a high charge/discharge rate capability (815 mAhg⁻¹ at 1000 mA g⁻¹) and long cycle life (>160 cycles). This strongly suggests that α -Fe₂O₃@C is a promising anode material for LIBs. More importantly, the method used in the synthesis of α -Fe₂O₃ nanorings can be easily extended to other iron oxides anode materials as a template.

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2. Experimental section

2.1. Synthesis method

The α -Fe₂O₃ nanorings were synthesized by a hydrothermal treatment of FeCl₃ with NaH₂PO₄ and Na₂SO₄ as additives [19]. In a typical experimental procedure, 259.53 mg FeCl₃, 1.66 mg NaH₂PO₄ and 6.25 mg Na₂SO₄ were dissolved in 80 mL of deionized water. The solution was turned into a Teflon-lined stainless steel autoclave for hydrothermal treatment at 220 °C for 40 h. After cooling the autoclave down to room temperature, the samples were collected by centrifugation, washed with deionized water and absolute ethanol for several times, and finally dried in air at 80 °C.

To synthesize α -Fe₂O₃@C nanorings, 0.25 g of the as-prepared α -Fe₂O₃ nanorings and 1.0 g of glucose were dispersed into 80 ml of deionized water. After under ultrasonic wave for 15 min, the solution was turned into a Teflon-lined stainless steel autoclave of 100 mL for hydrothermal treatment at 180 °C for 4 h. After the autoclave cooled down, the samples were washed with deionized water and absolute ethanol for several times, then dried in air at 80 °C for 10 h. α -Fe₂O₃@C nanorings were obtained by calcining above precipitate at 400 °C for 2 h in N₂ atmosphere.

2.2. Characterization

The power X-ray diffraction (XRD) measurement was taken by an XRD diffractometer (D8-Advance, Bruker, USA) using Cu K α radiation ($\lambda = 1.5418$ Å). Scanning electron microscopy (SEM) images were obtained through a field emission scanning electron microscopy (FESEM, SU70, Japan) using an accelerating voltage of 5 KV. TEM and HRTEM analysis were performed on a field-emission transmission electron microscope (TEM, JEOL, JEM2000, accelerating voltage 200 KV). Thermogravimetric (TG) curves were measured on an SDT Q600 thermal analyzer.

2.3. Electrochemical measurements

The electrochemical measurements were carried out using coin cells (CR2032). Active materials (α -Fe₂O₃ nanorings, α -Fe₂O₃@C nanorings), conductivity agent (carbon black), and binder (Carboxymethyl Cellulose, CMC) in a weight ratio of 80:10:10 were blended with deionized water as solvent. Electrode film was prepared by coating the slurry on a copper foil, then dried under

vacuum at 120 °C for 12 h. The sample loading for α -Fe₂O₃ and α -Fe₂O₃@C was 2.83 mg/cm² and 2.65 mg/cm², respectively. The coin cells were fabricated using Celgard 2400 microporous membrane as the separator, and LiPF₆ (1 M) in ethylene carbonate/dimethyl carbonate/diethyl carbonate (EC/DMC/DEC, 1:1:1 vol%) as the electrolyte. The assembly of the coin cells was carried out in an Ar-filled glovebox. After 12 h aging treatment, the cycle performance and rate capability of the cells were measured within a voltage window from 0.005 to 3.00 V (vs Li/Li⁺) on a battery testing system (LAND CT 2001A, China).

3. Results and discussion

3.1. Structure and morphology

Fig. 1 shows XRD spectra of α -Fe₂O₃ and α -Fe₂O₃@C nanorings. All the peaks in Fig. 1a can be indexed by the rhombohedral α -Fe₂O₃ phase (JCPDS no. 33-0664) without any peaks of other phases, indicating the high purity of as-prepared α -Fe₂O₃. Fig. 1b is the XRD pattern of the α -Fe₂O₃@C nanorings, which has a similar diffraction

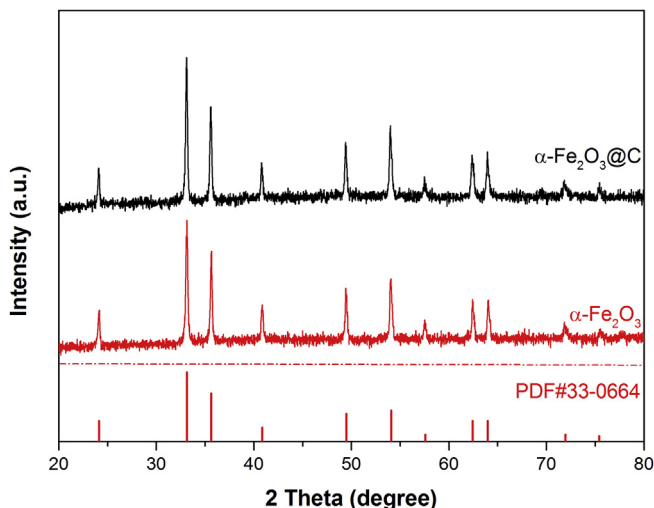


Fig. 1. XRD patterns of the α -Fe₂O₃ and α -Fe₂O₃@C nanorings. The identified peaks can be assigned to rhombohedral hematite unambiguously (JCPDS card no. 33-0664).

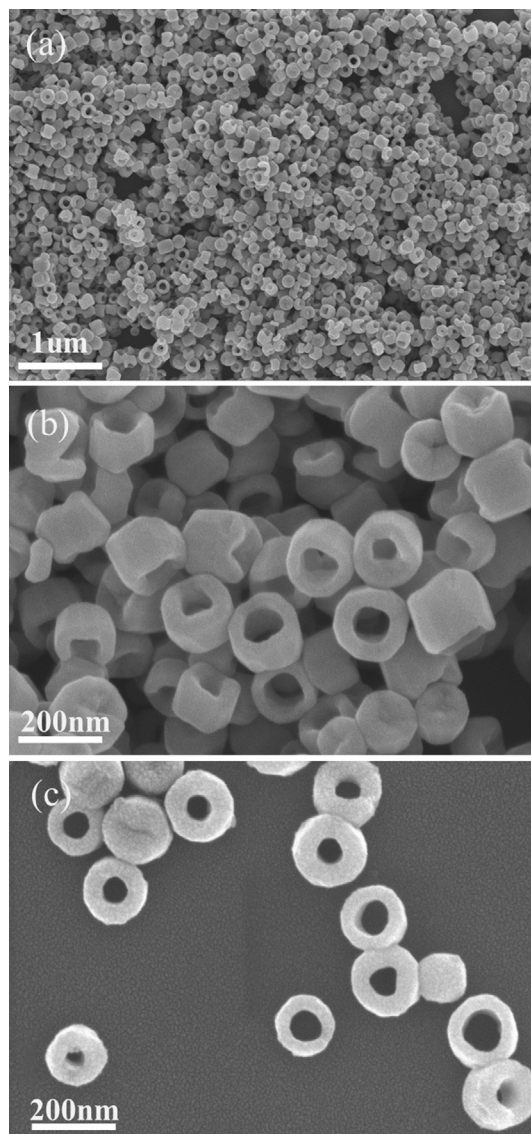


Fig. 2. (a,b) SEM images of α -Fe₂O₃ nanorings; (c) SEM images of α -Fe₂O₃@C nanorings.

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