



A simple preparation of rod-like Fe₂O₃ with superior lithium storage performance

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ABSTRACT

There are increasing interests in fabricating Fe₂O₃ anode materials in recent years owing to their high theoretical capacity and natural abundance. However, Fe₂O₃ anode materials encounter great challenges in association with their intrinsic poor electronic conductivity and drastic volume variation during cycling. Herein, rod-like Fe₂O₃ is prepared through a simple thermal decomposition method. When evaluated as anode material for LIBs, the rod-like Fe₂O₃ exhibits excellent rate capability (~400 mAh g⁻¹ capacity even at high current density of 7 A g⁻¹). Furthermore, high reversible capacity of ~500 mAh g⁻¹ can be retained at 3 A g⁻¹ after 1000 cycles. This work demonstrates that the thermal decomposition method is facile and efficient for the preparation of high-performance Fe₂O₃ anode materials for LIBs.

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1. Introduction

Lithium ion batteries (LIBs), have been widely applied in electric vehicles, portable electronics, and stationary energy storage devices due to their high energy density, environmental friendliness, and no memory effect compared to other secondary batteries [1–4]. Increasing requirements make it an emergency task for pursuing superior anode materials to replace conventional graphite-base anode. Transition metal oxides (TMOs) such as Fe₂O₃, NiO, and Co₃O₄ have drawn great attentions since Poizot et al. first reported that TMOs have electrochemical activity toward lithium [5]. Among those TMOs, Fe₂O₃ stands out for because of its high theoretical capacity, natural abundance, non-toxicity, and low cost [6,7]. However, the sluggish kinetics of conversion reaction, the intrinsic poor conductivity, and the severe pulverization of Fe₂O₃ electrode during discharge–charge process lead to poor cyclability and low rate capacity [8,9]. So far, various novel nanostructured Fe₂O₃, such as nanorods [10], nanotubes [11], hollow spheres [12], and nanosheets [13] have been fabricated to address these issues. Currently, many methods such as hydrothermal method, template method, and spray pyrolysis method were used to prepare these nanostructured Fe₂O₃, which showed improved electrochemical performance. However, the facile, efficient, low cost, and

large-scale production of these nanostructured electrode materials is still a big challenge.

In the present work, we demonstrate a rather facile, efficient, and low-cost strategy for fabricating hierarchical rod-like Fe₂O₃ material. The as-synthesized rod-like Fe₂O₃ exhibits superior high rate capability and outstanding cycling stability when applied as an anode material for LIBs.

2. Experimental section

2.1. Synthesis of the Fe₂O₃ rods

The rod-like Fe₂O₃ were synthesized by thermal decomposition method with FeCl₃·6H₂O and glucose as raw materials. First, a 10 mL aqueous solution containing 4 g FeCl₃·6H₂O and 1.4 g glucose were transferred to a porcelain boat and heat treated in a muffle furnace at 350 °C for 2 h with a temperature ramping rate of 5 °C min⁻¹ to form the Fe₂O₃ precursor. Second, the Fe₂O₃ precursor was heat treated in Ar atmosphere at 800 °C for 2 h with a temperature ramping rate of 5 °C min⁻¹ to obtain the final Fe₂O₃ rods.

2.2. Physical characterizations

The crystal structure of the as-prepared sample was characterized by X-ray diffractometer (XRD, Dutch PANalytica X'Pert3 power) with Cu Kα radiation at the scanning range of 5–90° with

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a scanning speed of 5° min^{-1} . The surface morphology of the as-prepared sample was observed by Field-emission scanning electron microscope (SEM, Japan, Hitachi SU-5000). The BET surface area of the as-prepared sample was determined from adsorption isotherms using a Micromeritics ASAP 2020 Surface Area Analyzer.

2.3. Electrochemical measurements

The working electrode was fabricated by mixing the as-prepared Fe_2O_3 rods, super P carbon black, and PVDF binder with a mass ratio of 7:2:1 in N-methyl-2-pyrrolidone (NMP) solvent to form slurry. The slurry was pasted onto copper foil with a blade and was dried at 80°C for 12 h under vacuum conditions. Electrochemical measurements were performed using coin-type half cells (CR 2016) assembled in the Ar-filled glove box (Mikrouna, China) with lithium metal as counter/reference electrode and polypropylene film (Celgard, 2400) as separator. The electrolyte was 1 M LiPF_6 dissolved in a mixture of ethylene carbonate (EC), dimethyl carbonate (DMC), and diethyl carbonate (DEC) with a volume ratio of 1:1:1. Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were carried out on a CHI 760E electrochemical workstation (Chenhua, China). In EIS measurement, the frequency ranged from 100 KHZ to 0.01 HZ and the AC amplitude was 5 mV. Galvanostatic discharge/charge measurements were performed by a multi-channel battery testing system (Neware BTS-5V/10 mA, China).

3. Results and discussion

Fig. 1(a) gives the XRD pattern of the as-prepared sample. All the diffraction peaks can be well indexed to $\alpha\text{-Fe}_2\text{O}_3$ (JCPDS 33-0664) [14]. The BET surface area of the as-prepared sample is

$6.3 \text{ m}^2 \text{ g}^{-1}$ (Fig. S1(a)). Fig. 1(b)–(d) give the SEM images of the as-prepared Fe_2O_3 sample. It can be seen that the Fe_2O_3 exhibits a rod-like morphology. The rods are composed of interconnected spherical particles.

Fig. 2(a) presents the CV curves of the Fe_2O_3 electrodes at a scan rate of 0.1 mV s^{-1} . In the first cycle, a sharp peak at $\sim 0.55 \text{ V}$ corresponds to the reduction of Fe^{3+} to Fe^0 and the formation of solid electrolyte interface (SEI) film [15,16]. In the subsequent cycles, the reduction peak decreases and shifts to higher potential; a reduction peak at $\sim 0.8 \text{ V}$ and an oxidation peak at $\sim 1.9 \text{ V}$ can be observed. Fig. 2(b) shows the discharge/charge curves of the Fe_2O_3 electrode at 0.5 A g^{-1} in various cycles. The extended discharge plateau at $0.8\text{--}0.7 \text{ V}$ and the subsequent slope in the range of $0.6\text{--}0.01 \text{ V}$ during the first discharge process are ascribed to the reduction of the Fe^{3+} to Fe^0 and the generation of the SEI layer and Li_2O , which matches well with the CV curves shown in Fig. 2(a). The voltage hysteresis between discharge and charge profiles initially increases in the first 100 cycles. In the 200th cycle, which suffer the most cycle numbers, the hysteresis between discharge and charge profiles gets smaller, which may originate from the particle pulverization (nano-grains formation) with the increase of the number of cycles [17]. Fig. 2(c) depicts the cycling performance of the Fe_2O_3 electrode at 0.5 A g^{-1} for 200 cycles. The initial discharge and charge capacities are 1816 and 1088 mAh g^{-1} , respectively. The large irreversible capacity loss during the first cycle mainly results from the formation of SEI layer [18,19]. The Fe_2O_3 electrode delivers a reversible capacity of 925 mAh g^{-1} after 200 cycles, which is superior to those previous reported Fe_2O_3 materials (Supporting information Table 1). Fig. 2(d) shows the rate capability of the Fe_2O_3 electrode at the current densities ranging from 0.5 to 7 A g^{-1} . A reversible capacity of 950 (in the seventh cycle),

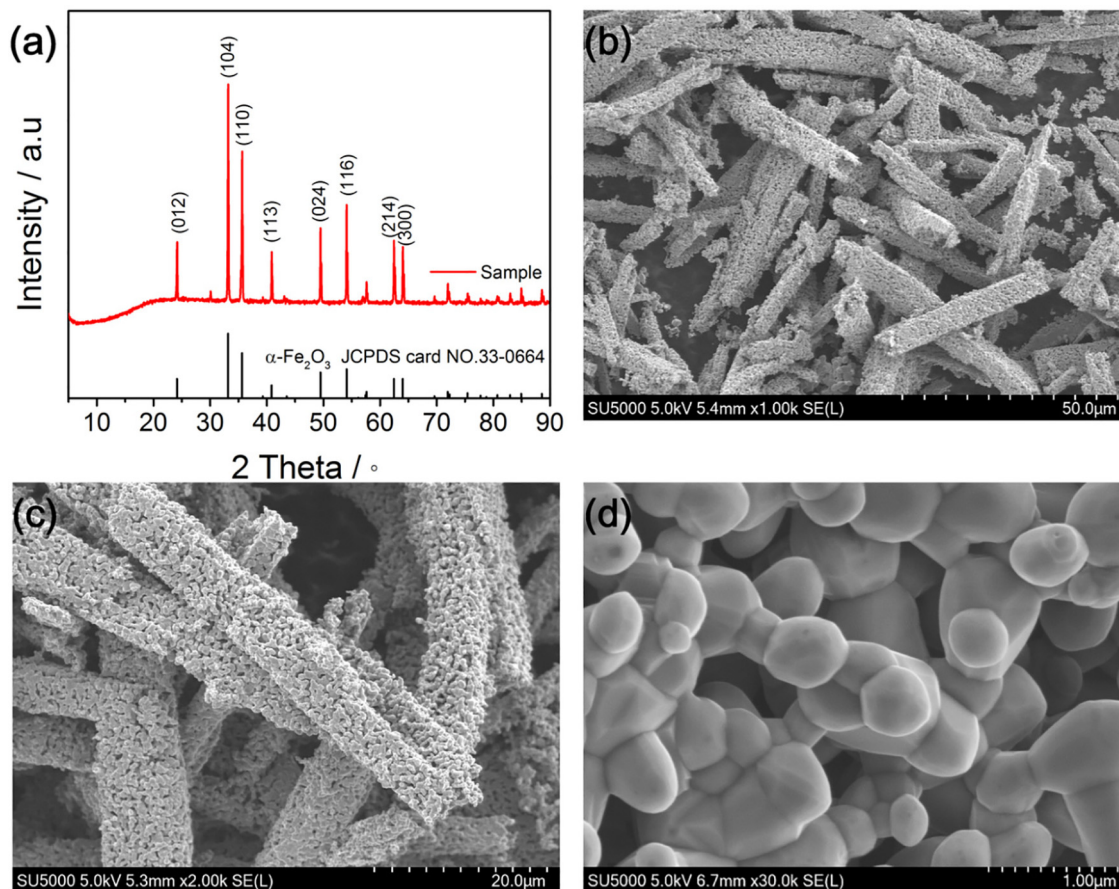


Fig. 1. (a) XRD pattern and (b)–(d) SEM images of the as-prepared Fe_2O_3 sample.

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