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Preparation and electrochemical properties of TiO₂/C/Si composites for lithium ion batteries



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ABSTRACT

TiO₂, C/Si and TiO₂/C/Si composite materials are synthesized with a simple hydrothermal method as anode materials for lithium ion batteries (LIBs). X-ray diffraction (XRD), Raman spectroscopy and field-emission scanning electron microscopy (FESEM) reveal that composite with C and TiO₂ does not influence the crystal structure of pristine Si. It is found that the TiO₂/C/Si composite exhibits enhanced capacity retention and excellent rate capability compared with Si, TiO₂ and C/Si composite. The composite delivers a high capacity of 861 mA h g⁻¹ at 0.1 C after 50 cycles. Even at high current density, the TiO₂/C/Si composite still demonstrates good cycling stability. The improvement in electrochemical performances can be attributed to the synergy effect of carbon and TiO₂, in which the carbon mixed naturally enhances the electrical conductivity and the TiO₂ outside effectively suppresses the volume change of Si particles.

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1. Introduction

Silicon has been recognized as an alternative anode material for lithium ion batteries (LIBs) and has drawn considerable attention since it has advantages of high theoretical capacity (4200 mA h g⁻¹), low insertion potential and high relative abundance. The huge volume change (300%) during repeated charge/discharge in cycling and the resulting short cycle life still hinder its widespread application [1–4]. Recently, great efforts have been made on material design, such as nanostructured Si [5] and Si-based composite electrodes [6]. Among these materials, C/Si composites are more attractive due to the conductive and ductile features of carbon, which is effective in improving conductivity and buffering the volume changes of Si during repeated charge/discharge [7,8]. Despite the substantial progress in improving the cycling performance of Si, the poor safety and performance issues still restrict the practical application of C/Si composite. As one of the nano-transition metal oxides, TiO₂ has attracted considerable attention for its many advantages such as low price, non-toxic, and pollution-free natures. Particularly, nano-TiO₂ has a little structure change in repeated charge/discharge, and demonstrates low polarization, good reversibility and safety performance [9,10].

In this present work, the TiO₂/C/Si composite was synthesized by a simple hydrothermal method. Satisfactorily, more improved cycling stability and rate capability compared with its pristine Si material are obtained due to the synergy effect of carbon and TiO₂.

2. Experimental

In a typical synthesis, 1 g of glucose and 1 g of urea were dissolved in 10 ml of deionized water and 25 ml of ethanol under mild stirring. Then 0.34 ml (1 mmol) of tetrabutyl titanate and 0.028 g (1 mmol) Si powder were successively added into the mixing solution with vigorous stirring. Afterwards, the obtained precursor was transferred into a Teflon-lined autoclave and heated at 180 °C for 24 h. The resulting slurry was then filtered and washed with deionized water before drying in a vacuum oven. Finally, the powder was heated at 500 °C for 5 h under flowing argon to obtain TiO₂/C/Si composite. The C/Si composite and TiO₂ particles were prepared using the same hydrothermal process.

X-ray diffraction (XRD) patterns were recorded using a DX-2700 diffractometer (Fangyuan, Dandong) with Cu K α radiation of $\lambda=0.154145$ nm. Raman measurements were performed using a Renishaw RM-1000 Raman spectrometer. The morphologies of samples were observed using a field-emission scanning electron microscopy (FESEM; JEOL, JSM-7001F).

For the fabrication of electrodes, 70 wt% of active material was mixed with 15 wt% of acetylene black and 15 wt% of binder

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(polyvinylidene fluoride, PVDF). 1 M LiPF₆ dissolved in a mixture of ethylene carbonate (EC) and diethyl carbonate (DEC) with the volume ratio of 1:1 was used as an electrolyte. The cells were charged and discharged using a battery tester (NEWARE, China) between 0 and 3.0 V (versus Li/Li⁺) at room temperature.

3. Results and discussion

Fig. 1(a) displays the XRD patterns of Si, TiO₂, C/Si and TiO₂/C/Si samples. All the diffraction peaks in the bottom two curves can be indexed to the pure phases of anatase TiO₂ (JCPDS 21-1272) and Si (JCPDS 65-1060). Not surprisingly, the diffraction peaks of both Si and TiO₂ are identified in the profiles of C/Si and TiO₂/C/Si. Nevertheless, no peak corresponding to carbon can be observed, which can be possibly attributed to its amorphous nature. Raman spectra are collected and shown to confirm the components of the samples (Fig. 1(b)). Si and TiO₂ display typical characteristic vibrational peaks in the four curves. For the C/Si and TiO₂/C/Si composites, two characteristic peaks centered at 1362 and

1585 cm⁻¹, which can be assigned to the D-band and G-band of carbon, providing direct evidence for the existence of carbon.

The surface morphologies of Si, TiO₂, C/Si and TiO₂/C/Si composites are presented in Fig. 2. The Si particles are irregularly shaped, as observed in Fig. 2(a). The as-prepared TiO₂ is homogeneously distributed as small particles with an average size of 40 nm (Fig. 2(b)). As shown in Fig. 2(c), the Si particles are dispersed in carbon matrix, in which the acute edges and corners of Si particles are easy to be distinguished. In Fig. 2(d), TiO₂/C/Si composite demonstrates quite similar morphology compared with that of TiO₂ in Fig. 2(b), in which the C/Si particles cannot be found. Taken the preparation process of TiO₂/C/Si composite into account, it is most possible that the lighter TiO₂ covers the C/Si composite totally. The element mapping images of TiO₂/C/Si composite conducted by energy-dispersive spectroscopy (EDS) were collected (not shown), which illustrate a homogeneous distribution of C and Ti in the TiO₂/C/Si composite. The thermogravimetric (TG) curve of TiO₂/C/Si composite was tested using a TA Q600 Thermal Analysis System to determine the content of carbon. Combined with the quantitative element analysis of EDS

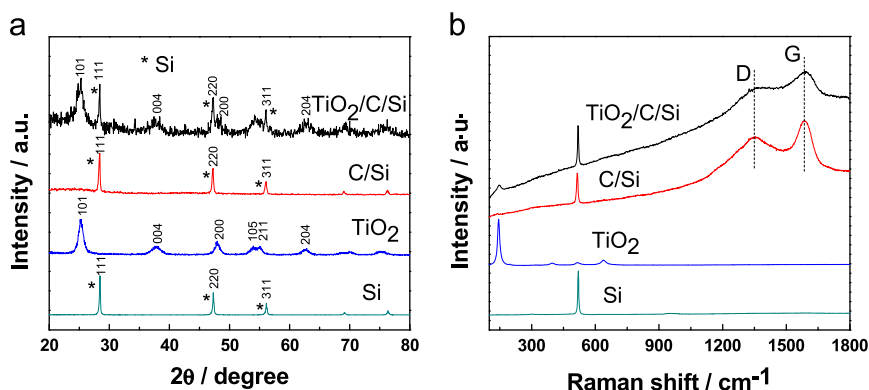


Fig. 1. XRD patterns (a) and Raman spectra (b) of Si, TiO₂, C/Si and TiO₂/C/Si samples.

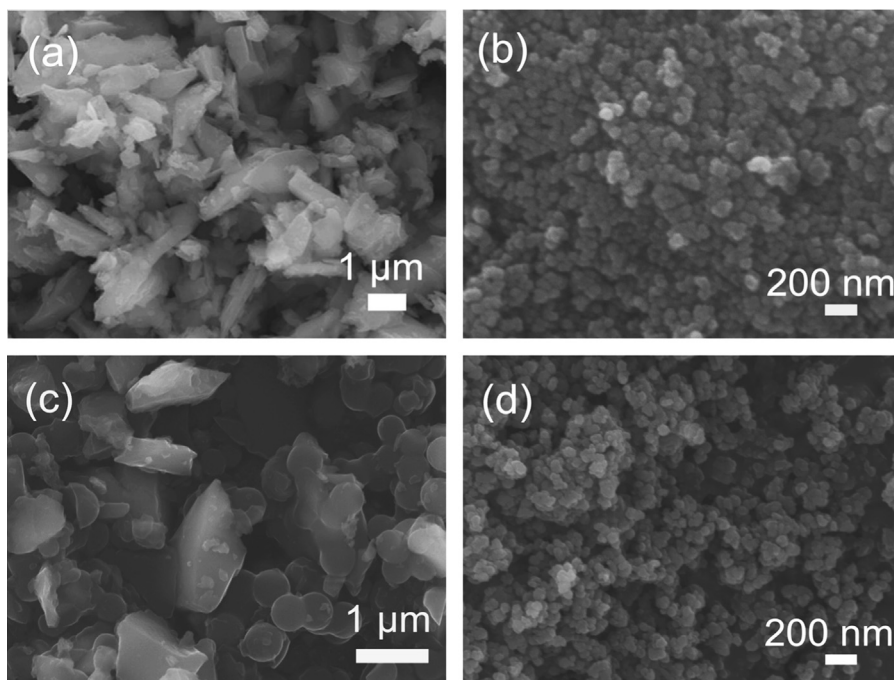


Fig. 2. FESEM images of Si (a), TiO₂ (b), C/Si composite (c) and TiO₂/C/Si composite (d).

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