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CNTs@ γ -Fe₂O₃@C composite electrode for high capacity lithium ion storage

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ABSTRACT

Electrode materials with high specific capacity and cycle stability are the key factors that determine the overall performance of lithium ion batteries (LIBs) for new smart electronic device, and such materials with high performance are still challenging. Here, we report a composite material Carbon nanotube @ Iron oxide @ Carbon (γ -CNTs@ γ -Fe₂O₃@C) with coaxial cable structure in hydrothermal method with the assistance of glucose. The as-prepared composite materials show superior electrochemical properties with good rate capability and excellent cycling performance, which show the great potential of CNTs@ γ -Fe₂O₃@C for practical production and application in energy devices.

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Introduction

With the development of smart equipment such as cellphones and electric/hybrid vehicles, the requirements for energy storage devices with high performance are also increasing [1–4]. Lithium ion batteries (LIBs) are most popular choices because of their moderate energy density, long life span, and environmental friendliness [5–7]. In spite that, a LIB is composed of a number of components, and the electrode materials are the key factor that fundamentally determines its performance, especially its capacity and cycle performance [8,9].

Recently, there has been large number of reports on the one-dimensional (1-D) nanomaterials as the LIB electrode materials, due to their unique advantage of being able to buffer a significant amount of volume variations, which makes them especially suitable for high-capacity LIB anode applications compared with their conventional 3-D analogs [10–12]. Among the various 1-D materials, carbon nanotubes (CNTs), which possess open network for short diffusion length and high electronic conductivity, are quite favorable for mass transportation and electrochemical conversion of lithium ions, making them the most popular anode candidates in research [13–16]. For the pristine CNTs samples, there is a

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more severe agglomeration of CNTs and the consumption of electrolytes is also higher due to the formation of the SEI in the first charging process. This makes the pristine CNTs material suffer from lower specific capacity & initial coulombic efficiency when used as an electrode material alone, which significantly limits the practical application of CNTs to a large extent.

To tackle with this issue, a simple but effective strategy is to composite CNTs with various secondary materials, which possess a much higher specific capacity than pristine CNTs. This gives solution to multiple issues for both the CNTs and the secondary materials. On the one hand, it provides sufficient electronic conductivity for the secondary materials, which are often semi-conductive or even insulating. On the other hand, it makes the overall capacity of the CNT-based composites much higher than the pristine CNTs. Besides, the open network in the composite provides additional bonus for the large volume change buffering and facile mass diffusion. By far, a series of secondary materials have been utilized for this purpose, including CNTs-supported transition metal sulfides (Co_{1-x}S , NiS , MoS_2 , etc) [17], non-metallic oxide (TiO_2 , SiO_2 , etc) [18], transition metal oxides (Fe_3O_4 [19], SnO_2 [9,20], Co_3O_4 [21] and NiO [22], etc) and so on.

Among these materials, the transition metal oxides are the most frequently studied materials because of their high theoretical capacity and relatively low cost [4]. And a series of technologies have been developed to integrate these materials with CNTs, including directly mixing these two components [21,22], vapor/liquid phase deposition of the secondary component on the CNTs [23], or thermal pyrolysis of the mixture of their precursors [24]. Apart from these, additional carbon coating has also been applied and proven effective to improve the electrochemical stability of these materials [25], by using different precursors including glucose, oleic acid, ethylene glycol, and ionic liquids [26–28]. Such carbon layers have also been believed to further decrease the aggregation, volume expansion, and collapse during the cycling, and provide rapid and continuous electronic transport at the same time [29].

Therefore, it would be highly desirable to design such CNTs@metal oxide@C with a coaxial cable structure, in which the synergistic effect of the carbon cushion of the coated layers and the high electronic conductivity of the hosts is expected to simultaneously enhance the electrochemical performance of the nanocomposites. However, the materials with such merits are still very rare and the preparation of similar-structured materials often involved very complex and multiple-stepped processes, making them economically unviable [30,31]. Thus, the development of a facile synthetic route to generate a hybrid metal oxide coaxial material with superior energy storage performance from metal oxide and inner CNT/outer carbon layer is of great scientific interest and practical necessity.

Herein, we report a 1-D CNTs@ $\gamma\text{-Fe}_2\text{O}_3$ @C material with a special coaxial cable structure through a hydrothermal reaction and a subsequent heat treatment process, for lithium ion battery anode application. The as-prepared materials exhibit a high initial reversible capacity of 1360 mAh g^{-1} at current density of 50 mA g^{-1} , with an initial coulombic efficiency of 68.1% and near 100% in the subsequent cycles.

Moreover, it exhibits excellent cycling performance with a retention about 97.67% after 150 cycles at a very high current density of 2 A g^{-1}

Experiment

Materials synthesis

Synthesis of CNTs@ $\text{Fe}_5\text{O}_7(\text{OH})\cdot 4\text{H}_2\text{O}$ @C

CNTs@ $\text{Fe}_5\text{O}_7(\text{OH})\cdot 4\text{H}_2\text{O}$ @C nanocables were synthesized by using the hydrothermal method with glucose. In a typical procedure, 40 mg of CNTs powder (multiwalled carbon nanotubes, 98%, carbon >90%, Chinese Academy of Science), 40 mg of CTAB ($\text{C}_{16}\text{H}_{33}(\text{CH}_3)_3\text{NBr}$, 98.5%, Tianjin Jiangtian Chemical Technology Co., Ltd), 0.485 g of $\text{Fe}(\text{NO}_3)_2$ (98.5%, Tianjin Jiangtian Chemical Technology Co., Ltd), 0.6 g of urea ($\text{CH}_4\text{N}_2\text{O}$, AR, Tianjin Guangfu Fine Chemical Research Institute) and 0.16 g of glucose ($\text{C}_6\text{H}_{12}\text{O}_6$, 98.5%, Tianjin Jiangtian Chemical Technology Co., Ltd) are dissolved into 25 ml ethanol and 50 ml deionized water. After being ultrasonically dispersed for 30 min, the solution was then transferred into a Teflon-lined stainless-steel autoclave with a capacity of 100 ml and kept at $190 \text{ }^\circ\text{C}$ for 12 h. When the autoclave was naturally cooled down to room temperature, the black products were collected, which then went through the repeated centrifugation and washed with deionized water and dried at $60 \text{ }^\circ\text{C}$ for 6 h. The as-prepared material was denoted as CNTs@ $\text{Fe}_5\text{O}_7(\text{OH})\cdot 4\text{H}_2\text{O}$ @C.

Synthesis of CNTs@ $\gamma\text{-Fe}_2\text{O}_3$ @C

The preparation of CNTs@ $\gamma\text{-Fe}_2\text{O}_3$ @C was a heat treatment process. The CNTs@ $\text{Fe}_5\text{O}_7(\text{OH})\cdot 4\text{H}_2\text{O}$ @C powder was transferred into a quartz boat and placed in a tube furnace under argon atmosphere. The material was maintained at $600 \text{ }^\circ\text{C}$ for 2 h with a beforehand heating rate of $5 \text{ }^\circ\text{C min}^{-1}$, and the furnace was then naturally cooled down to room temperature when the reaction was finished. For comparison purpose, the amount of glucose was controlled at 0.12, 0.16, 0.20 and 0.24 g to control the thickness of the carbon layer outside the matrix. Here, the corresponding products was denoted as Fe-1, Fe-2, Fe-3 and Fe-4, respectively.

Materials characterizations

The crystal structure was characterized by X-ray diffraction method (Rigaku D/Max 2500 v/pc). Specific surface area and pore size distribution of the products were measured using a nitrogen adsorption-desorption analyzer (NOVA 2200e surface area analyzer, Quantachrome, USA). TG/DSC measurement was taken with a thermogravimetric analyzer (SDT Q600 V20.9 Build 20). Field emission scanning electron microscopy (FESEM) images were obtained on a Hitachi S-4800 microscope. Transmission electron microscopy (TEM) images were taken on a JEOL JEM-2100F microscope. X-ray photoelectron spectroscopy (XPS) studies were performed using monochromatic Al K α radiation (Axis Ultra, Kratos, 150 W, 15 kV and 1486.6 eV), and the binding energies of the samples were calibrated by taking the carbon 1s peak as the reference (284.6 eV).

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