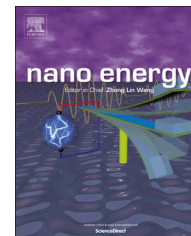




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RAPID COMMUNICATION

Silica-assistant synthesis of three-dimensional graphene architecture and its application as anode material for lithium ion batteries



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Abstract

A new approach for scalable synthesis of three-dimensional graphene (3d-G) with the assistant of silica is reported. Silica is generated on the surface of graphene oxide through a facile solution reaction. Graphene coated by silica is reduced through thermal reduction. 3d-G can be obtained after the subsequent etching process. The structures and morphologies of samples are investigated. It is demonstrated that the agglomeration of individual graphene sheets during the thermal reduction is effectively avoided under the perfect protection of silica. The electrochemical studies of the material show a significant enhancement in the specific capacity and rate capability of the lithium ion battery over commercially available graphite electrodes due to its unique 3D cross-linked graphene architecture. This method enables us to develop a facile and effective approach to scalable preparation of graphene.

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Introduction

Lithium ion batteries have been widely used as energy storage devices for portable electronic devices, and they are identified as one of the most important and the greenest type of rechargeable batteries [1–3]. In state-of-the-art energy technologies, high energy density lithium ion batteries are in great demand to satisfy the requirement of portable electronic devices and full electric vehicles.

The energy density and performance of lithium ion batteries largely depend on the physical and chemical properties of the electrode materials [4]. At present, graphitic carbon anodes are often employed in lithium ion batteries which can void the problem of Li dendrite formation, guarantee good cyclability and safety [5]. However, the theoretical specific capacity of graphite is only 372 mA h g^{-1} [6]. In order to meet the increasing demand for lithium ion batteries with high energy density, many efforts have been made to explore new anode materials [7-10].

Since the discovery of graphene in 2004, the emergence of graphene nanosheets has opened up an exciting field in the science and technology of two-dimensional (2D) nanomaterial due to its exceptional properties [11-16]. Graphene is an atomically thick, 2D nanosheet which is composed of sp^2 carbon atoms arranged in a honeycomb structure. Graphene can offer great advantages owing to its chemical stability, high elasticity, good conductivity, extremely high carrier mobility, and superior mechanical strength [17,18]. Particularly, graphene has superior electrical conductivity than graphitic carbon, high theoretical surface area of over $2600 \text{ m}^2 \text{ g}^{-1}$, chemical tolerance and a broad electrochemical window that would be very favorable for application in energy technologies [19-21]. Graphene nanosheet was first obtained by micromechanical exfoliation of graphite to produce high-quality graphene with superior structure, but the yield of this method is extremely low and the process is hard to control [22]. Chemical vapor deposition and epitaxial growth can also obtain graphene with few layers and large area, such as the decomposition of ethylene on nickel [23]. To date, the most promising strategy for low cost and large scale synthesis of graphene is via graphene oxide (GO) route due to the scalability [24]. In this method, GO can be reduced by hydrazine, dimethylhydrazine, NaBH_4 , as well as thermal and solvothermal reduction [25,26].

With respect to lithium ion batteries, it is believed that owing to its large surface-to-volume ratio and highly conductive nature, graphene may have properties that make it suitable for reversible lithium ion storage in lithium ion batteries. However, the availability of 2D graphene is hindered by the spontaneous agglomeration and restacking of graphene nanosheets, resulting from the high cohesive van der Waals attractions between the neighboring sheets [27]. The aggregation of graphene results in significant degradation of the unique properties of individual sheets, such as high specific surface area, fast ion transport behaviors and the large lithium storage on both sides of graphene surface [19,28]. To amplify the intrinsic properties of graphene, numerous approaches have been explored to address the stacking issue. Doping is an effective solution, which often introduces nanoparticles, carbon nanotubes or polymers as the spacer into the interlayer spaces to obtain graphene-based 3D hybrid materials [29-31]. 3D graphene architecture are of great interest in energy, catalysis, sensors and biological fields due to the desirable properties, such as flyweight, high porosity, large specific area and high electrical conductivity [32-35]. Recently, there have been some reports on the preparation of 3D graphene in forms of aerogels or foams. For example, Gao et al. reported the synthesis of ultra-flyweight graphene-based carbon aerogels by freeze-drying aqueous solutions of CNTs and GO followed

by chemical reduction process [36]. 3D graphene foam is typically synthesized by chemical vapor deposition using nickel foam as the template [37-39]. In these approaches, the existence of spacers and the relatively low production hindered the wide application of graphene. So, it is still a challenge to prepare 3D graphene architecture on a considerable scale in the absence of chemical cross-linkers or spacers.

In this work, we have prepared the spacer-free 3D graphene architecture (3d-G) with a step-by-step synthesis route. Silica layers were firstly fabricated on the surface of GO via the hydrolysis of tetraethylorthosilicate. The resulting powder was then pyrolyzed under nitrogen atmospheres to form a sandwich-like G/SiO_2 sheets. A further etching process was adopted to remove SiO_2 layers to fabricate the final 3d-G architecture. During this procedure, the bending and crumpling resulted in 3D graphene architecture without aggregation. For comparison, reduced GO (rGO) samples obtained through the reduction of hydrazine were synthesized. While evaluating the electrochemical performance of 3d-G as anode material for lithium ion batteries, it exhibits a large reversible capacity of 659 mA h g^{-1} at a rate of 0.5 C as well as good cycling performance and good rate capability, demonstrating 3d-G a promising candidate for anode material in lithium ion batteries.

Experimental details

Materials

Graphite powder (purity > 99.99%) is purchased from Nanjing Xianfeng Nano Technology Co. Sulfuric acid (95-98%, AR grade), sodium nitrate (AR grade), potassium permanganate (AR grade), hydrogen peroxide (30%, AR grade), hydrochloric acid (36-38%, AR grade), sodium hydroxide (AR grade), ammonium hydroxide (25-28%, AR grade), hydrazine (80%, AR grade) and hydrofluoric acid (40%, AR grade) are from Xilong Chemical Reagent Co. Cetyltriethylammonium bromide (CTAB), tetraethoxysilane (TEOS) are obtained from Sinopharm Chemical Reagent Co. All reagents are used as received.

Preparation of GO/SiO_2

Graphite oxide was synthesized from graphite powder based on the modified Hummer's method [24]. As-prepared graphite oxide was dispersed in deionized water by ultrasonication for 2 h to get the homogeneous GO aqueous dispersion. SiO_2 layers were generated on the surface of cationic surfactant modified GO via the hydrolysis of TEOS. In a typical experiment, 0.2 g of NaOH and 4 g of CTAB were dissolved in GO dispersion under moderate magnetic stirring. 4 ml of TEOS was added drop by drop under stirring at 35°C . The mixture was further stirred for 10 h. The resulting brown precipitate was collected by vacuum filtration, washing thoroughly with ethanol and deionized water, and then drying.

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