Three-dimensional SnO$_2$ nanoparticles synthesized by joule heating as anode materials for lithium ion batteries

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Abstract

Tin dioxide (SnO$_2$) is a promising material for use as anodes because of its high theoretical capacity (1,494 mAh g$^{-1}$). However, a critical limitation is the large change in volume during repeated cycling by pulverization of SnO$_2$, which results in capacity fading. In this study, we enhanced cycle life and reduced capacity fading by introducing the use of three-dimensional SnO$_2$ nanoparticles on carbon nanofibers (CNFs) as an anode material, which is fabricated by simple carbothermal shock through the Joule heating method. Our observations show that the SnO$_2$ nanoparticles are about 50 nm in diameter and are uniformly distributed on CNF, and that the strong connections between SnO$_2$ nanoparticles and CNF are sustained even after repeated cycling. This structural advantage provides high reversible capacity and enhanced cycle performance for over 100 cycles. This study provides insight into the fabrication of anode materials that have strong electric connections between active materials and conductive materials due to the Joule heating method for high-performance lithium ion batteries.

1. Introduction

Development of lithium ion batteries (LIBs) with high energy densities is required because of the ever-growing demand for large-scale energy storage applications such as electric vehicles (EVs) and hybrid EVs [1–3]. High-capacity anodes are important since the limited capacity of anode materials is a main bottleneck in current LIB systems. Among diverse anode candidates, tin dioxide (SnO$_2$) has been widely studied because of its high theoretical specific capacity, safe working potential, low cost, and environmental friendliness [4–7]. The principal reaction mechanism of SnO$_2$ as an anode material involves the two-step electrochemical process [8]:

\[ \text{SnO}_2 + 4\text{Li}^+ + 4e^- \leftrightarrow \text{Sn} + 2\text{Li}_2\text{O} \] (1)

\[ \text{Sn} + x\text{Li}^+ + xe^- \leftrightarrow \text{Li}_x\text{Sn} \quad (0 \leq x \leq 4.4) \] (2)

On the basis of conversion and alloying reactions, SnO$_2$ has a high specific capacity of 1,494 mAh g$^{-1}$, which is advantageous for achieving high energy densities.

However, the practical application of SnO$_2$ is limited as it becomes pulverized during alloying and dealloying due to high volume expansions of around 300%. Repeated cycling accelerates the pulverization of SnO$_2$, which causes the loss of electrical contact and formation of an unwanted solid electrolyte interphase (SEI). Consequently, the LIB will have a low Coulombic efficiency (CE) and poor cyclic performance.

Much effort has been made to address these problems. Nanostructuring of SnO$_2$, such as particle size reduction to the nanoscale [9], thin-film formation [10], pore generation [11, 12], and hierarchical structuring [13–15], can effectively prevent pulverization by accommodating volume expansion. In addition, the shortened
diffusion distance and enlarged surface area of nanostructured SnO$_2$ allow lithium ions to move more freely and provide more active sites for the reactions. Furthermore, compositing SnO$_2$ with carbonaceous materials such as carbon nanotubes [16], carbon nanofibers (CNFs) [17], graphene [18–20], and reduced graphene oxides [21] can effectively improve electrochemical performance [22–24]. The high electrical conductivity of carbon-based materials enhances electron transfer in the anode. Moreover, carbon can serve as a scaffold for supporting SnO$_2$ in the composites, which prevents the agglomeration of nanoparticles and the formation of unwanted SEI layers. Specifically, the porous structure of carbon scaffolds can alleviate volume changes by effectively accommodating lithium during repeated alloying and dealloying [25, 26]. Thus, a composite anode material comprising nanoscale SnO$_2$ with porous carbon can be highly preferable. However, previous methods mostly use wet chemistry to prepare SnO$_2$ nanoparticles and their composite structure with carbon. Although wet chemistry approaches allow the shape, size, and composition of synthesized nanoparticles to be controlled, it is difficult to uniformly incorporate SnO$_2$ nanoparticles on carbon with a high mass fraction.

We propose a new strategy for fabricating SnO$_2$ nanoparticles on CNF for anodes in LIBs by using a straightforward approach involving carbothermal shock (CTS) through Joule heating. SnO$_2$ nanoparticles can be efficiently fabricated on the CNF surface by Joule heating for very short durations (＜1 s). This achieves high coverage of the prepared SnO$_2$ with mass fractions up to 80 wt%. Our study will provide new insight into the use of CTS through Joule heating for fabrication of anode materials, and can solve the adhesion issue of SnO$_2$. The method is significantly simple and quick by applying the Joule heating for very short durations. Furthermore, the uniformly distributed and strongly connected SnO$_2$ nanoparticles uniformly incorporated on carbon with a high mass fraction.

2. Methods

2.1. Preparation of polyacrylonitrile (PAN) nanofibers (NFs)

Polymer solution for electrospinning is prepared by dissolving 1 g of PAN (150,000 Mw, Sigma-Aldrich) in 6.5 g of N, N-dimethylformamide (99.8%, Sigma-Aldrich) with stirring at 70 °C for overnight. For electrospinning, the prepared polymer solution was injected by syringe and a voltage of 15 kV was applied between the collector plate and the metal nozzle. The feeding rate was 10 µl min$^{-1}$ and the distance between the collector and the nozzle was 15 cm. After the electrospinning, the freestanding PAN sheet (white color) was detached from the collector. The freestanding PAN sheet was stabilized at 280 °C for 1 h and changed to brown color. The stabilized PAN sheet should be annealed at high temperature for 2 h with Ar (300 sccm). The annealing temperature was adjusted for joule heating because the resistances of the carbon nanofiber sheet depend on the annealing temperature.

2.2. Joule heating for synthesis of SnO$_2$ nanoparticles on CNFs

The annealed PAN film (carbon nanofiber, CNF) was used as the carbon substrate for the Joule heating. For the Joule heating, the CNF film was cut into 2 mm × 20 mm size and attached to Cu electrode using silver paste. The precursor solution (600 mM SnCl$_2$ in ethanol) was dropped onto the CNF film and dried on the hot plate (60 °C for 30 min). After drying, the CNF was placed in the customized vacuum chamber, and an external power supply was connected to the Cu electrodes. With Ar purging (30 sccm), a current pulse (1 A for 1 sec) was applied to the CNF for the Joule heating.

2.3. Characterizations

An x-ray powder diffractometer (Rigaku Ultima IV diffractometer) with a graphite monochromator equipped with a Cu Kα line (40 Kev/40 Ma) was used to observe the crystalline structure of the synthesized anode materials. The morphology of pristine, lithiated, and delithiated anodes were characterized by SEM (Tescan Mira 3 LMU FEG, 20 kV). Particle size measurement and elemental analysis were performed using TEM (Tecnai G2 T-20S, FEI & JEM-3011 HR, Jeol) equipped with EDS.

2.4. Electrochemical measurements

The SnO$_2$ nanoparticles on CNF were used as an anode. Li metal foil and polypropylene membrane were used as a reference/couter electrode and a separator, respectively. The electrolyte was prepared by dissolving 1 M lithium hexafluorophosphate (LiPF$_6$) in a mixture of ethylene carbonate (EC) and dimethyl carbonate (DMC) (1:1, volume ratio), with 10 wt% fluoroethylene carbonate (FEC), and 2 wt% vinylene carbonate (VC). For comparison, SnO$_2$ nanoparticles (Sigma-Aldrich) were fabricated into anodes by mixing SnO$_2$, Super P, and poly(vinylidene fluoride) (PVdF) (8:1:1, weight ratio) in N-methyl pyrrolidone, and the resulting slurry was coated on a piece of copper foil. All the process of assembling the cell was carried out in an Ar-filled glove box using CR2032-type coin cells. The loading mass of SnO$_2$ was 5 mg cm$^{-2}$. The galvanostatic charge–discharge
cycling was carried out by a battery cycler (Toscat-3100, Toyo Co. Ltd). The anode was lithiated using constant currents of 0.1 C followed by a constant voltage (CC/CV) at 0.05 V. Delithiation was then performed using constant currents of 0.1 C with a cut-off voltage of 2.5 V. CE was calculated from the ratio of the delithiated capacity to the lithiated capacity. The internal resistance was calculated by using galvanostatic intermittent titration technique (GITT), which applies constant current of 0.1 C for 30 min followed by a relaxation for 1 h. Electrochemical impedance spectroscopy (EIS) was conducted using Bio-Logic with a frequency range from $10^5$ to 0.1 Hz at an amplitude of 10 mV.

3. Results and discussion

In this study, the CTS method first reported in 2018 was used to synthesize SnO$_2$ nanoparticles [27]. In this highly effective method, metal precursors on the carbon substrate surface are melted and mixed by passing an electric current into the carbon substrate for a very short duration (<1 s) and then raising and cooling the temperature instantaneously. We selected this method because it is a simple approach for producing small nanoparticles and mixing various metals. Figure 1 is a schematic of the CTS process for SnO$_2$ nanoparticle synthesis. CNF was used as the carbon substrate to prepare SnO$_2$ nanoparticles. CNF is one of the most widely used carbon materials in electrochemical applications because of its conductive and porous properties. We fabricated the CNF film used in this study by electrospinning, and its average diameter was about 500 nm. Sn nanoparticles were first made prior to the synthesis of SnO$_2$ nanoparticles. A 600 µl volume of 50 mM SnCl$_2$ dissolved in ethanol was dropped on the CNF film. Next, Joule heating was conducted by passing a 1 A current through the CNF film. This caused the temperature of the CNF film to rapidly rise to a high degree for 600 ms. Subsequently, the temperature cooled rapidly and nanoparticles formed on the CNF surface. The nanoparticles were naturally oxidized to SnO$_2$ after 24 h under ambient conditions (25 °C air temperature).

Figure 2 shows the morphology of the SnO$_2$ nanoparticles on the CNF. Scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy-dispersive x-ray spectroscopy (EDS) mappings were conducted to investigate the morphology and distribution of SnO$_2$ particles, and results are shown in figures 2(a) and (b). The high-magnification SEM image shows that the spherical SnO$_2$ nanoparticles were attached well on the CNFs. The SnO$_2$ nanoparticles were about 50 nm in diameter and were uniformly and fully distributed over the surface of CNFs without disrupting the fibrous structure. The formation of SnO$_2$ nanoparticles on CNFs was clearly visible in the TEM/EDS images (figure 2(b)). The SnO$_2$ particles were 50 nm in diameter, which is in good agreement with the SEM results. The SnO$_2$ content of the total anode material was analyzed by thermogravimetric analysis (figure S1). The CNF underwent complete decomposition after
calcination under an oxidative atmosphere at 800 °C, showing a mass change of 20%. Thus, the remaining SnO₂ content was estimated to be 80 wt%. X-ray diffraction (XRD) analysis was conducted to verify the formation of SnO₂ on CNFs, and the results are shown in figure S2. The XRD pattern shows typical diffraction peaks at 25.6°, 33.8°, and 51.8°, which are indexed to the cassiterite (tetragonal rutile) structure of SnO₂ (JCPDS no. 04-003-0649) [28, 29]. To compare with the SnO₂ on CNF that was prepared by Joule heating, spherical SnO₂ nanoparticles were purchased, and crystalline and morphological analyses were performed (figure S3). The crystal structure revealed by XRD exhibited a cassiterite structure, and the particle diameter was on the scale of tens of nanometers in the TEM images. Meanwhile, the valence of SnO₂ for two samples was examined by using XPS analysis, which is provided in figure RX. The Sn 3d₅/₂ spectra in both samples contain an intense peak at 486.8 eV which is assigned to Sn⁴⁺, indicating the presence of SnO₂ in both samples [30].

Galvanostatic charge–discharge tests were performed to investigate differences in lithiation and delithiation behaviors between SnO₂ nanoparticles and the Joule-heating-derived SnO₂ nanoparticles on CNF (figure 3). Morphological changes were monitored during the pristine, lithiation, and delithiation stages. There were several electrode cracks in the SnO₂ nanoparticles after the first cycle (figure 3(a)), indicating that electric conductive paths in the SnO₂ nanoparticles were disrupted because of the high volume changes during the lithiation and delithiation reactions. In contrast, the SnO₂ nanoparticles derived from Joule heating were uniformly distributed and strongly connected to CNF, thereby providing electric conductive paths, as shown in figure 3(a) (pristine). Notably, the connections between the SnO₂ nanoparticles and the CNF remained after lithiation and delithiation.

Figures 3(b) and (c) show the voltage profiles of Li cells containing the SnO₂ nanoparticles or the SnO₂ nanoparticles on CNF, at 1, 2, 5, 10, and 20 cycles. In the first delithiation, the reversible capacity of SnO₂ nanoparticles on CNF was large (910 mAh g⁻¹), while that of the SnO₂ nanoparticles was comparatively low (360 mAh g⁻¹). Moreover, the reversible capacity of SnO₂ nanoparticles on CNF remained large in the following cycles, while the capacity of the kind decreased sharply to almost 0 mAh g⁻¹ in the 20th cycle. The high reversibility of capacity in SnO₂ nanoparticles on CNF can be attributed to strong connections and electric conductive paths between the SnO₂ nanoparticles and the CNF. During charge and discharge, the SnO₂ nanoparticles undergo large volume changes, which lead to electrode deformation. In these adverse conditions, a lack of strong connections between the SnO₂ nanoparticles and conductive agents would accelerate electrode deformation and promote the generation of electrically dead particles. A loss of electrical contact with SnO₂ nanoparticles leads to decreased reversible capacity and capacity fading. In contrast, the strong connections between SnO₂ nanoparticles and CNF provide stable electric conductive paths during charge and discharge and result in a high reversible capacity.

Figures 4(a) and (b) show the capacity retention and CE of Li cells with the SnO₂ nanoparticles or the SnO₂ nanoparticles on CNF at a current density of 0.1 C rate. The cells with SnO₂ nanoparticles on CNF exhibited enhanced capacity retention after 100 cycles (figure 4(b)), while we observed sharp capacity fading in the cell with the nanoparticles (figure 4(a)). Moreover, the CE of Li cells with SnO₂ nanoparticles on CNF was higher and more stable than that of the SnO₂ nanoparticles. The superior cycle stability of the SnO₂ nanoparticles on CNF can be attributed to the strong connections between SnO₂ nanoparticles and CNF, as previously discussed. During repeated cycling, the SnO₂ particles undergo large volume changes and generate dead particles. This results in a loss of electric conductive paths, as well as electrical insulation by polymeric SEI, as shown figure 4(c).
Capacity fading occurs as the dead particles are unable to react. However, for the SnO2 nanoparticles on CNF, electric conductive paths are maintained even after long-term cycling, as shown in figure 4(d). Hence, the cells with SnO2 nanoparticles on CNF exhibit high reversibility of capacity and excellent cycle performance without sharp capacity fading. The reaction mechanisms in both samples are briefly shown in figures 4(e) and (f). As mentioned above, for the SnO2 nanoparticles/Li cells, the electric conductive paths between the SnO2 particles can be easily disconnected by massive volume change during the lithiation/delithiation since the SnO2 particles are simply mixed with the PVdF binder and the carbon conductive agent (figure 4(e)). However, for the SnO2 nanoparticles on CNF, the SnO2 particles are strongly connected to CNF by Joule heating. Thus, the electric conductive paths between the SnO2 particles and CNF are hardly disconnected even though the SnO2 particles undergo repeated volume expansion and contraction during the cycling (figure 4(f)). Consequently, the strong connections between the SnO2 particles and CNF derived by Joule heating are an important key factor for cycle improvement. In addition, the improved cycle performance at a current density of 1 C (figure S5a) and higher capacity at various current densities of 0.2 C, 0.5 C, 1 C and 2 C (figure S5b) of the Li cells with the SnO2 nanoparticles on CNF support the effect of the strong connections between the SnO2 particles and CNF. The reduced resistance of the cells with SnO2 nanoparticles on CNF can be ascribed to the strong connections (figure S6, S7).

4. Conclusions

As a new strategy for making SnO2 anode materials, SnO2 nanoparticles on CNF was fabricated by CTS through Joule heating, and its electrochemical performance for LIBs was investigated. The SnO2 nanoparticles on CNF exhibited enhanced cycle life without sharp capacity fading after 100 cycles. This improvement in
electrochemical performance can be attributed to strong connections and electric conductive paths between SnO$_2$ nanoparticles and CNF. Analyses using XRD, TEM, and SEM revealed that these nanoparticles were around 50 nm in diameter and were uniformly distributed on CNF, and the strong connections between SnO$_2$ nanoparticles and CNF were stable even after long-term cycling. We expect that this study will provide inspiration for the fabrication of anode materials by using simple dry method. Most previous works have used the wet method for the fabrication of carbon composite anode materials, which needs a lot of time to fabricate the materials and the morphology control is very difficult. However, our method is very simple and quick by applying the Joule heating. Moreover, the carbothermal shock can generate the uniformly distributed SnO$_2$ nanoparticles on CNF with very strong connections. Thus, these favorable features of our method can open up a new strategy for the fabrication of carbon composite anode materials.

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**Data availability statement**

All data that support the findings of this study are included within the article (and any supplementary files).
Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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