Introduction

A supercapacitor as an effective energy-storage device is usually classified as an electric double-layer capacitor and a pseudocapacitor. Carbon fiber (CF) has attracted significant attention due to its high stiffness, high tensile strength, low weight, and high chemical inertness. However, one challenging aspect of a CF electrode material is how to increase the active sites for driving electrochemical reactions since the electron transfer process occurs only at the interface between the solid electrode and the aqueous or gel electrolyte. Surface modification would be an efficient strategy to improve the electrochemical properties of CF. Pseudocapacitive materials such as metal oxides and conductive polymers are well introduced into CF. Electroactive materials are capable of rapid reversible redox reactions occurring on the CF surface, which could well enhance the capacitive performance of CF composite electrode. Transition metal compounds have been investigated in detail as electroactive supercapacitor electrode materials. Tin dioxide (SnO₂) is an n-type semiconductor with a wide band gap of 3.6 eV and a low electron affinity. SnO₂ has a high theoretical capacity of 780 mAh g⁻¹, attracting considerable attention in supercapacitor applications. However, the weak electronic conductivity of SnO₂ has restrained its applications in high-performance energy-storage systems. SnO₂ composites using conductive materials usually show improved capacitance performance. SnO₂/CFs have been studied in detail as electroactive electrode material of lithium-ion batteries. Doping of SnO₂ with appropriate elements

Synthesis and electrochemical performance of an electroactive nitrogen-doping SnO₂ nanoarray supported on carbon fiber

Yibing Xie

Abstract

An electroactive nitrogen-doping tin dioxide nanorod array (N-SnO₂ NRA) is designed as an effective energy-storage electrode material for supercapacitor applications. N-SnO₂ supported on a carbon fiber substrate is prepared using SnCl₄ as a precursor through hydrolysis, hydrothermal growth, and an NH₃-nitriding process. Electroactive N-SnO₂ is formed by an N-doping reaction between Sn(OH)₄ and NH₃, revealing a high nitrogen-doping level of 12.5% in N-SnO₂. N-SnO₂/carbon fiber reveals a lower ohmic resistance and charge transfer resistance than SnO₂/carbon fiber, which is consistent with its higher current response and lower voltage drop in electrochemical measurements. N-SnO₂ NRA has an independent nanoarray structure and a small side length of a quadrangular nanorod, contributing to a more accessible interspace, reactive sites, and feasible electrolyte ion diffusion. The N-SnO₂/carbon fiber NRA electrode shows higher specific capacitance (105.4 F g⁻¹ at 0.5 A g⁻¹) and rate capacitance retention (45.0% from 0.5 to 5 A g⁻¹) than a SnO₂/carbon fiber NRA electrode (58.6 F g⁻¹, 38.4%). Significantly, the cycling capacitance retention after 2000 cycles increases from 78.1% of SnO₂/carbon fiber to 98.8% of N-SnO₂/carbon fiber, presenting a superior electrochemical cycling stability. The N-SnO₂ supercapacitor maintains stable power working at an output voltage of 1.6 V. The specific capacitance decreases from 75.2 to 55.1 F g⁻¹ when the current density increases from 1 to 10 A g⁻¹. The corresponding energy density decreases from 24.23 to 9.81 Wh kg⁻¹, presenting a reasonable rate capability. So, the prepared N-SnO₂ nanorod array demonstrates superior capacitance performance for energy-storage applications.

Keywords
Carbon fiber, capacitance, nitrogen-doping, supercapacitor, tin dioxide

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can directly change its electronic structure and effectively modulate its electronic properties, accordingly improving the capacitance properties for supercapacitor applications.\textsuperscript{31,32} Nitrogen has been proposed as a good p-type dopant to improve the electronic conductivity of SnO\textsubscript{2} and enhance the electrochemical capacitance contributed by reversible Faradaic reactions. It is reported that N-doped SnO\textsubscript{2} subsequently has higher electrochemical performance than pristine SnO\textsubscript{2}.\textsuperscript{33–35} Experimental and theoretical investigations on N-doped SnO\textsubscript{2} also demonstrate consistent results for the improvement of electrochemical properties.\textsuperscript{36}

In this study, nitrogen-doping tin dioxide nanorod array (N-SnO\textsubscript{2} NRA) is designed, fabricated, and supported on a CF substrate, forming a novel N-SnO\textsubscript{2}/CF NRA electrode.\textsuperscript{28,37} The N-SnO\textsubscript{2}/CF NRA electrode with high nitrogen-doping and an independent nanoarray structure improves the conductivity and electroactivity. The N-SnO\textsubscript{2}/CF NRA electrode is expected to have high electrochemical performance. This N-SnO\textsubscript{2}/CF NRA electrode is also used to construct an N-SnO\textsubscript{2} supercapacitor, while maintaining high compatibility with the proton acid polymer gel electrolyte in solid-state supercapacitor applications for effective energy storage.

**Results and discussion**

**Preparation process**

Figure 1(a) shows the schematic illustrating the preparation process of N-SnO\textsubscript{2}/CF electrode. The N-SnO\textsubscript{2} NRA supported on CF substrate is prepared using SnCl\textsubscript{4} as a precursor through a hydrolysis process, a hydrothermal process, and an NH\textsubscript{3}-nitriding process. Figure 1(b) shows a photograph of the SnO\textsubscript{2}/CF and N-SnO\textsubscript{2}/CF NRA electrodes. Both the SnO\textsubscript{2}/CF and N-SnO\textsubscript{2}/CF NRA electrodes present a colorful appearance when they are grown on CF through the hydrothermal process. Comparatively, the N-SnO\textsubscript{2}/CF NRA electrode presents slighter gray appearance compared to the SnO\textsubscript{2}/CF NRA electrode, which is related to the nitriding treatment of SnO\textsubscript{2} NRA. A solid-state asymmetric N-SnO\textsubscript{2} supercapacitor was then constructed using the N-SnO\textsubscript{2}/CF NRA electrode as a positive electrode, TiN/CF as a negative electrode, and H\textsubscript{2}SO\textsubscript{4}-polyvinyl alcohol as a gel polymer electrolyte.

**Morphology and structure characterization**

Figure 2 shows scanning electron microscope (SEM) images of SnO\textsubscript{2} NRA and N-SnO\textsubscript{2} NRA. Both SnO\textsubscript{2} and N-SnO\textsubscript{2} adopt a uniform nanorod array with quadrangular prism shape. The side length of the quadrangular nanorod is 50–60 nm for SnO\textsubscript{2} and 20–30 nm for N-SnO\textsubscript{2}. Comparatively, N-SnO\textsubscript{2} shows a bigger interspace of neighboring nanorods than SnO\textsubscript{2}, accordingly promoting the electrolyte ion diffusion in the electrochemical reaction. Figure 3 shows the energy diffraction X-ray spectra of SnO\textsubscript{2} and the N-SnO\textsubscript{2} nanorod array. Table 1 lists the elemental analysis of SnO\textsubscript{2} and the N-SnO\textsubscript{2} nanorod array. SnO\textsubscript{2} comprises tin (21.4 at %) and oxygen (78.6 at %). The atomic ratio of O/Sn is much larger than 2:1, which is ascribed to the surface hydroxylation group of SnO\textsubscript{2}. The nitrogen, oxygen, and tin elements are determined in N-SnO\textsubscript{2}, which indicates that the nitrogen has been doped into SnO\textsubscript{2}. A high oxygen atomic percent of 72.3% is also due to the surface hydroxyl group. The relative nitrogen atomic percentage is 12.5%, indicating a high nitrogen-doping level in N-SnO\textsubscript{2} NRA. Usually, Sn(OH)\textsubscript{4}\textsuperscript{−} is formed by hydrolysis of the SnCl\textsubscript{4} precursor which is shown as the reaction formula (1). SnO\textsubscript{2} is formed by dehydration annealing treatment of Sn(OH)\textsubscript{4}\textsuperscript{−} as shown as the reaction formula (2). N-SnO\textsubscript{2} is prepared by nitriding treatment of SnO\textsubscript{2}, which is shown as the reaction formula (3). Comparatively, Sn(OH)\textsubscript{4}\textsuperscript{−} has a higher reactivity than SnO\textsubscript{2} with NH\textsubscript{3} due to the superior interfacial proton acid-base affinity. Accordingly, a high nitrogen-doping level is achieved by nitriding treatment of Sn(OH)\textsubscript{4}\textsuperscript{−}, which is shown as the reaction formula (3). Sufficient nitrogen-doping could lead to improving the conductivity and the reactivity of N-SnO\textsubscript{2}. It should be noted that the N-doping content is not optimized in N-SnO\textsubscript{2} for supercapacitor applications. The formation of N-SnO\textsubscript{2} (SnO\textsubscript{2}−xNx) involves the following reactions.

\begin{equation}
\text{SnCl}_4 + 4\text{NaOH} \rightarrow \text{Sn(OH)}_4 + 4\text{NaCl}
\end{equation}

\begin{equation}
\text{Sn(OH)}_4 \rightarrow \text{SnO}_2 + 2\text{H}_2\text{O}
\end{equation}

\begin{equation}
2\text{Sn(OH)}_4 + 4\text{NH}_3 \rightarrow 2\text{SnO}_2\cdot x\text{N}_x + (6 - 2x) \text{H}_2 + (4+2x) \text{H}_2\text{O} + (2 - x) \text{N}_2
\end{equation}

\begin{equation}
2\text{SnO}_2 + 4\text{NH}_3 \rightarrow 2\text{SnO}_2\cdot x\text{N}_x + (6 - 2x) \text{H}_2 + 2x \text{H}_2\text{O} + (2 - x) \text{N}_2
\end{equation}

Figure 4 shows the X-ray diffraction (XRD) patterns of SnO\textsubscript{2}/CF and N-SnO\textsubscript{2}/CF. Concerning the CF substrate, SnO\textsubscript{2}/CF and N-SnO\textsubscript{2}/CF exhibit a strong XRD peaks at around 20 = 25°, corresponding to the (002) crystal plane of graphitic carbon. Both samples show similar characteristic diffraction peaks at 26.6°, 33.8°, and 38.0°, which could be indexed to the (110), (101), and (200) crystal planes of tetragonal rutile SnO\textsubscript{2} (JCPDS card no. 41-1445). N-SnO\textsubscript{2}/CF does not show other new characteristic diffraction peaks. So, N-SnO\textsubscript{2} and SnO\textsubscript{2} exhibit similar crystal phase. Figure 5(a) shows the survey X-ray photoelectron spectroscopy (XPS) spectra of SnO\textsubscript{2} and N-SnO\textsubscript{2}. As common points, the characteristic peaks of Sn 3p, Sn 3d, Sn 4p, Sn 4d, and O 1s are ascribed to the SnO\textsubscript{2} in both samples. The characteristic peaks of C 1s are ascribed to the CF substrate. Considering the different points, N-SnO\textsubscript{2}/CF NRA demonstrates an obvious characteristic peak due to N 1s. It confirms the N-doping effect on SnO\textsubscript{2}. Figure 5(b) shows the N 1s XPS peaks of N-SnO\textsubscript{2}. The dominant peak at 398.5 eV is assigned to the Sn -N bond, which involves the nitrogen-substitution of O sites in bulk SnO\textsubscript{2} and the surface tin oxynitride species at the edge of SnO\textsubscript{2}. It is believed that the N-doping causes the formation of the nitrogen dopant sites and oxygen vacancy.
defects in SnO$_2$.\textsuperscript{38,39} It is also reported that the N 2p gap states are induced by an N-substituting O site, which results in a narrowed band gap.\textsuperscript{40} This effect accordingly contributes to enhancing the electrical conductivity of N-SnO$_2$. The results of the XPS studies are in good agreement with the results of energy-dispersive X-ray spectroscopy (EDX) characterization.

**Electrochemical properties**

Figure 6 shows the electrochemical impedance spectra of the SnO$_2$/CF and N-SnO$_2$/CF NRA electrodes and the corresponding equivalent circuit. The equivalent circuit elements include ohm resistance ($R_\text{o}$), charge transfer resistance ($R_{\text{ct}}$), and the constant phase element (CFE) and the Warburg impedance element ($W_\text{f}$). The ohm resistance can be obtained from the intercept of the impedance curve on the real axis in high-frequency region. Table 2 lists the fitting parameters of the equivalent circuit of the SnO$_2$/CF and N-SnO$_2$/CF NRA electrodes. The $R_\text{o}$ value decreases from 2.774 $\Omega$ for SnO$_2$ NRA to 2.141 $\Omega$ for N-SnO$_2$ NRA, indicating that the improved conductivity of N-SnO$_2$ contributed to by nitrogen-doping. The charge transfer resistance can be estimated from the semicircle diameter in
high-frequency region. The $R_{ct}$ value also obviously decreases from $1.24\ \Omega$ for SnO$_2$ NRA to $0.85\ \Omega$ for N-SnO$_2$ NRA, indicating the improved charge transfer of N-SnO$_2$ contributed to by the high interspace of the nanorods. The straight line in the low-frequency region is related to the diffusion-controlled Warburg impedance. The N-SnO$_2$ NRA shows a larger slope than the SnO$_2$ NRA, indicating better approaching an ideal capacitor performance. The CFE can be defined by the capacitance (CFE-T) and the constant phase element exponent (CFE-P). N-SnO$_2$ NRA shows a higher CFE-T (1.968) than SnO$_2$ NRA (1.312), indicating its more porous structure. N-SnO$_2$ NRA shows a relatively higher CFE-P (0.963) than SnO$_2$ NRA (0.894), indicating that it is closer to being an ideal capacitor. The $W_0$ value can be defined by the Warburg phase element exponent ($W_0^{-P}$), the time constant ($W_0^{-T}$), and the Warburg resistance ($W_0^{-R}$). Both SnO$_2$ and N-SnO$_2$ NRA show similar $W_0^{-P}$ values of around 0.5, indicating the finite-length Warburg diffusion. N-SnO$_2$ NRA shows a much lower $W_0^{-R}$ ($0.719$) than SnO$_2$ NRA ($3.164$), indicating that it is more suitable for electrolyte ion diffusion. N-SnO$_2$ NRA shows a relatively lower $W_0^{-T}$ ($0.020$) than SnO$_2$ NRA ($0.023$), indicating a higher effective diffusion coefficient.

Figure 7(a) and (b) shows the cyclic voltammetry (CV) curves of the SnO$_2$/CF and N-SnO$_2$/CF NRA electrodes at different scan rates. The CV curves of both electrodes maintain a similar shape without obvious distortion at different scan rates from 5 to 200 mV s$^{-1}$. Figure 7(c) shows the response current curves at different scan rates. Herein, the average response current density is defined as $I = \left( \int_{V_c}^{V_a} i(V) \, dV \right) / \Delta V$, where $V_a$ and $V_c$ represent the lowest and the highest potential (mV), respectively. Obviously, N-SnO$_2$/CF reveals a higher average response current density than SnO$_2$/CF at the same scan rate, indicating its improved conductivity and electroactivity. This result is consistent with the comparison result of the $R_{ct}$ value. Figure 7(d) and (e) shows the galvanostatic charge and discharge (GCD) curves at different current densities. The GCD curves show the symmetric characteristic between the charge and discharge counterparts. When the current density increases from 0.5 to 5 A g$^{-1}$, SnO$_2$/CF NRA shows a specific capacitance decrease from 58.6 to 22.5 F g$^{-1}$, presenting the rate capacitance retention of 38.4%. N-SnO$_2$/CF NRA shows the specific capacitance decreases from 105.4 to 47.4 F g$^{-1}$, presenting a rate capacitance retention of 45.0%. So, N-SnO$_2$/CF NRA presents higher capacitance and higher rate capability than SnO$_2$/CF NRA. Comparatively, N-SnO$_2$/

| Table 1. Elemental analysis of SnO$_2$ and N-SnO$_2$ nanorod array. |
|----------------|----------------|
| Element | SnO$_2$ | N-SnO$_2$ |
| O   | 78.6%  | 72.3%  |
| Sn  | 21.4%  | 15.2%  |
| N   | 0%     | 12.5%  |

Figure 3. Energy diffraction X-ray spectra of (a) SnO$_2$ and (b) N-SnO$_2$.
CF NRA also demonstrates a higher capacitance performance than as-reported different SnO$_2$ electrode materials. Figure 7(e) and (f) shows the cycling capacitance retention curves of the SnO$_2$/CF and N-SnO$_2$/CF NRA electrodes. The cycling capacitance retention after 2000 cycles increases from 78.2% for SnO$_2$/CF NRA to 98.8% for N-SnO$_2$/CF NRA.
representing a superior electrochemical cycling stability of the N-SnO₂/CF NRA electrode.

Figure 8(a) and (b) shows the potential-extended CV curves at 5 mV s⁻¹ and the potential-extended GCD curves at 1 A g⁻¹ for N-SnO₂ supercapacitor. When the window potential is extended from 0.8 to 1.6 V, the CV and GCD curves of the N-SnO₂ supercapacitor mostly keep a similar shape without any obvious distortion or deformation. The polarization effect becomes more and more obvious at a higher potential above 1.6 V. Hence, subsequent electrochemical measurements of the N-SnO₂ supercapacitor were controlled at 1.6 V. Figure 8(c) and (d) shows the CV curves at scan rates of 5–200 mV s⁻¹ and GCD curves at current densities of 1–10 A g⁻¹ for the N-SnO₂ supercapacitor. The CV curves maintain a similar rectangle shape without any obvious distortions from 5 to 200 mV s⁻¹. Also, the GCD curves remain in a rectangular shape without any obvious distortions from 1 to 10 A g⁻¹. The specific capacitance decreases from 75.2 to 55.1 F g⁻¹, while keeping the high rate capacitance retention of 73.3%. This means that the N-SnO₂ supercapacitor can conduct a stable and reversible charge–discharge process at an output voltage of 1.6 V.

Figure 8(e) shows the Ragone plot of the N-SnO₂ supercapacitor. When the current density increases from 1 to 10 A g⁻¹, the power density increases from 800 to 8000 W kg⁻¹. Meanwhile the energy density decreases from 24.23 to 9.81
Wh kg\(^{-1}\), while maintaining the high-performance capacity in comparison with other reported SnO\(_2\) supercapacitors.\(^{42}\) Moreover, the N-SnO\(_2\) supercapacitor also demonstrates higher energy density than an as-reported different SnO\(_2\) supercapacitor.\(^{43}\) Figure 8(f) shows photographs of the N-SnO\(_2\) supercapacitor powering electric devices. It should be noted that the N-SnO\(_2\) supercapacitor is composed of a single unit without any serial or parallel connection. It can well power a light-emitting diode (LED) light bulb with a nominal voltage of 1.8 V and drive an electric fan with a nominal power of 1.0 W, indicating the high performance of the N-SnO\(_2\)/CF NRA electrode and its effective interaction with the H\(_2\)SO\(_4\)-PVA gel electrolyte. Hence, the N-SnO\(_2\) NRA demonstrates superior energy-storage capability.

**Conclusion**

An electroactive N-SnO\(_2\) NRA is designed and fabricated as an effective energy-storage electrode material for supercapacitor applications. N-SnO\(_2\) supported on a CF substrate is prepared using SnCl\(_4\) as the precursor through hydrolysis, hydrothermal growth, and an NH\(_3\)-nitriding process. N-SnO\(_2\) has a high nitrogen-doping level of 12.5% which exhibits highly improved conductivity. The N-SnO\(_2\)/CF NRA electrode shows higher specific capacitance and rate capacitance retention (105.4 F g\(^{-1}\) at 0.5 A g\(^{-1}\) and 45.0% from 0.5 to 5 A g\(^{-1}\)) than an SnO\(_2\)/CF NRA electrode (58.6 F g\(^{-1}\) and 38.4%). Moreover, the cycling capacitance retention after 2000 cycles increases from...
78.1% for SnO₂/CF to 98.8% for N-SnO₂/CF, presenting superior electrochemical cycling stability. The N-SnO₂ supercapacitor demonstrates stable and reversible charge–discharge operation at an output voltage of 1.6 V. When the current density increases from 1 to 10 A g⁻¹, the specific capacitance decreases from 75.2 to 55.1 F g⁻¹ and the energy density decreases from 24.23 to 9.81 Wh kg⁻¹. Hence, N-SnO₂ NRA demonstrates the superior capacitance performance.

**Experimental**

*Preparation of the N-SnO₂/CF electrode and N-SnO₂ supercapacitor*

The CF was immersed in 30 mL of 0.055 M SnCl₄ solution. 30 mL of 0.11 M NaOH solution was added dropwise in SnCl₄ solution and kept for 3 h at room temperature. Sn(OH)₄ colloidal particles were prepared through a hydrolysis process and were fully adsorbed onto the CF substrate, forming Sn(OH)₄/CF nanoparticles. A tin precursor solution was prepared from 30 mL of 0.0125 M SnCl₄ and 0.25 M NaOH aqueous solutions. The Sn(OH)₄/CF and this precursor solution were placed into a Teflon-lined stainless-steel autoclave. The hydrothermal process was conducted at 180 °C for 12 h. The obtained product was rinsed thoroughly with distilled water and dried in a vacuum oven at 60 °C to give the Sn(OH)₄/CF nanorod array. The Sn(OH)₄/CF was annealed in a tubular furnace at 400 °C for 4 h under a continuous ammonia vapor flow of 10 standard-state cubic centimeter per minute (sccm). An argon flow was used to cool down the sample, thus forming the N-SnO₂/CF nanorod array. For comparison, the obtained Sn(OH)₄/CF was subsequently converted into crystalline SnO₂/CF by annealing at 320 °C for 2 h in a Muffle furnace. The mass loading of SnO₂ and N-SnO₂ was 4.2 and 3.8 mg cm⁻¹ in the CF substrate, respectively.

*Characterization and measurement*

The surface microstructures and morphologies of SnO₂ and N-SnO₂ were characterized using an SEM (Zeiss Ultra Plus) under different magnifications. EDX (Oxford ISIS 310) was used to identify the element composition and N-SnO₂. XPS (ESCALAB 250) was used to analyze the elements and the chemical states of the materials. The XPS peaks were calibrated against the C 1s signal of the contaminant carbon at a binding energy of 284.6 eV. A CHI760D electrochemical workstation with a three-electrode configuration was used to conduct electrochemical measurements such as CV, GCD, and electrochemical impedance spectroscopy (EIS). It includes SnO₂/CF or N-SnO₂/CF as the working electrode, a Pt plate as the counter electrode, saturated calomel electrode (SCE) as the reference electrode, and 1.0 M H₂SO₄ as the electrolyte solution. EIS measurements were conducted under open-circuit potential conditions in a frequency range of 10⁻² to 10⁷ Hz by applying an alternating current (AC)-potential amplitude of 5 mV. The mass specific capacitance (C) according to GCD, energy density (E), and power density (P) of the obtained electrodes and device can be calculated using the following equations (5)–(7)

\[
C = \frac{I \Delta t}{m \Delta V} \tag{5}
\]

\[
E = \frac{C(NV)^2}{2} \tag{6}
\]

\[
P = \frac{1}{2}mV \tag{7}
\]

where \(I\) is the current density (A g⁻¹), \(\Delta t\) is the discharge time (s), \(\Delta V\) is the potential window (V), and \(m\) is the mass of the electroactive material in the electrode.

**Declaration of conflicting interests**

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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