



Solution Blown Silicon Carbide Porous Nanofiber Membrane as Electrode Materials for Supercapacitors



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ABSTRACT

Silicon carbide materials have attracted much attention in supercapacitor as electrode materials. Solution blowing process is a novel method to fabricate nanofibers with high productivity. In this study, silicon carbide nanofiber membranes (SiC-NFMs) with porous architecture were prepared via solution blowing process and subsequent calcination, which exhibiting good electrochemical performance as electrode materials for supercapacitors. As a simple and efficient nanofiber fabricating method, solution blowing was introduced here to produce as-spun precursor nanofiber membranes which would be pyrolysed to Silicon carbide products. The porous architecture of SiC-NFMs, which was composed of mesopores in each single nanofiber and macropores between nanofibers, led to the excellent electrochemical performance. The results showed that these membranes were of high specific capacitance ($\sim 189 \text{ F g}^{-1}$) and outstanding cycling stability (91.7% retention after 3000 cycles). This solution blowing method would find application to produce SiC-NFMs which can be used as electrode materials for supercapacitors with good performance.

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1. Introduction

Supercapacitors, also known as electrochemical capacitors (ECs) or ultracapacitors, are important electrical energy storage systems having wide applications in many areas such as memory back-up systems, portable electronic devices, transportation, and industrial renewable energy management [1–4]. Carbon-based nanomaterials, including activated carbon [5], carbon nanotubes (CNTs) [6], carbon nanowires (CNWs) [7], carbon nanofibers (CNFs) [8], and graphene [9], have been widely used as electrode materials for supercapacitors due to their high surface area, reasonable conductivity and chemical/thermal stability [10,11]. However, carbon-based nanomaterials are hydrophobic, and hence reduce electrode wettability, which might restrict them from being widely applied [12–14]. There are generally two methods to solve this problem. One method is to fabricate C/M/MOx mixed electrodes by

adding metal or metal oxides, which can improve capacitance resulted from the redox reaction during charging/discharging process [15–20]. The other method is to find some feasible materials with high capacity and long-term stability [21–23].

Silicon carbide (SiC) is known to be a wide-band gap semiconductor material with excellent physicochemical stability and thermal-mechanical properties. One dimensional (1D) SiC architectures (e.g., nanowires, nanorods, nanotubes, and nanofibers) have promising applications in catalyst supports, electronics, energy storage, filters and separators due to their unique properties [24–28]. Recently, some publications revealed that the micro-supercapacitors composed of 1D SiC nanomaterial electrodes showed long cycle life and high capacitance retention. Tee et al. synthesized nanostructured carbide-derived carbons (CDC) from SiC powders (SiC-CDC) via gas phase chlorination, and the specific capacitance was up to 130 F g^{-1} [29]. Yan et al. also synthesized the nanoscale microporous carbide-derived carbon (nano-CDC) by chlorination of SiC nano-powder, which specific capacitance was up to 141 F g^{-1} and almost 100% or more specific capacitance could be remained even after 10000 cycles [30]. Kim et al. prepared the micro and mesoporous silicon carbide flakes (PSF) by one-step carbonization of Si flakes. The PSF electrode oxidized for 24 h

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(OPSF–24 h) exhibited a high charge storage capacity, showing a specific capacitance of 243.3 F g^{-1} at a scan rate of 5 mV s^{-1} with 85.6% rate performance from 5 to 500 mV s^{-1} in 1.0 M KCl aqueous electrolyte [31]. However, to our best knowledge, there are few literatures to report the continuous SiC nanofiber membranes using for the supercapacitor. Electrospinning is a universal method for fabricating various organic and inorganic materials including SiC nanofiber. Eick et al. prepared SiC nanofibers by pyrolysis of electrospun preceramic polymers successfully [32]. Liu et al. also fabricated uniform SiC nanofibers with a diameter ranging from 1 to 2 nm through the technique of concentric electrospinning [33]. Nevertheless, most published studies about electrospinning were conducted by using a single-needle setup, and the nanofibers were obtained in a low yield, which may limit the industrial progress of fibrous nonwovens or membranes. In our previous work, SiC porous nanofiber membranes with outstanding thermal stability had been successfully prepared by solution blowing process [34], whereas the electrochemical properties of SiC nanofiber electrodes with well-designed pore structure have not been studied.

Herein, as an effective technique combining the advantages of electrospinning and dry spinning [35], SiC precursor nanofiber membranes were successfully produced via solution blowing process. During this process, the precursor polymer solution is stretched into many fine filaments by a high pressure airflow, then deposit randomly on the collector forming a three-dimensional nanofiber membrane or mat. Solution blowing process is of great potential for its simple operation, high production rate (fiber weight, 2.0–10.0 g/h per needle), universality in processing most polymers, ability to control parameters and versatility in producing multi structural and functional nanofibers [36–39].

The polycarbosilane (PCS) and polystyrene (PS) blended polymer solution was selected to prepare the as-spun nanofibrous membrane. PCS was a kind of linear polymer and commonly used as SiC precursor with good dimensional stability during calcination, and PS was added to improve the spinnability of the blended solution. After high temperature calcination, PS would decompose to be CO_2 and CH_4 , directly leading to the pore structure of each single SiC nanofiber [40–42]. Pore size on the SiC nanofiber surface could be well controlled by adjusting the proportion of PS and PCS, which played a key role in the formation of micro/mesopores and consequently its effect on electrochemical properties.

In this study, we demonstrated the production of SiC porous nanofibrous membrane simply by pyrolysis of the as-spun nanofiber membrane containing precursor polymer and a thermoplastic polymer. Solution blowing process was used to fabricate as-spun PCS/PS blended 3D nanofibrous membrane. Morphologies, structures, and specific surface areas of the as-spun nanofibers and the resultant SiC porous fibers were examined in detail. Moreover, the effects of the pore structure and size on electrochemical properties were tested via a three-electrode measurement system.

2. Experimental

2.1. Materials

Polycarbosilane (PCS; $M_w = 1,400$) was purchased from Cerafil Co. Ltd., Suzhou, China, and used as precursor. Polystyrene (PS; $M_w = 1,000,000$) was supplied by Aladin Co. Ltd., Shanghai, China, and used to improve the spinnability. Xylene (AR grade) was provided by Guangfu Regent Co. Ltd., Tianjin, China. All of the materials and reagents were used as received.

2.2. Preparation of Silicon Carbide Porous Nanofiber Membranes

The spinning solution was prepared by dissolving PCS and PS powder in the xylene with stirring for about 2 h. Then, the solution

was feed into the needle through a syringe pump. After the streams were pressed out of the needle, they were broken up into multistrand jets and were stretched to the extreme extent by the high-velocity gas flow. The as-spun nanofibers formed after solvent evaporation and deposited on a nonwoven collector. The detailed spinning conditions were listed in Table 1. Subsequently, a non-melting treatment (190°C for 2 h, in air) was performed to stabilize the fiber structure, and then these nanofibers were heated up to 1400°C with a rate of $2^\circ\text{C}/\text{min}$ in Nitrogen and maintained for 4 h under 1400°C . After the furnace cooled back to room temperature, the product was collected, washed, and vacuum dried.

2.3. Characterization

The appearances of samples were photographed by a digital camera (Sony, Japan), and 3D thickness distribution pattern of the nanofibrous membrane was obtained under an industrial optical microscope. The morphologies of samples were examined by field emission scanning electron microscopy (FE-SEM, S-4800, Hitachi Co., Japan) after 2 min gold coating treatment (E1045, Hitachi ion sputter, Japan), and the diameters of the samples were measured (100 times per sample) by image analyzer (ipwin32, Soft Imaging System). A JEM-2010 transmission electron microscope (TEM, JEOL, Japan) was also used to observe the morphologies and structures of the materials at 200 kV. X-ray diffraction (XRD) patterns were obtained on a X-ray diffractometer (XRD) (D8 Discover with GADDS, BRUKER AXS Co., USA) carrying out at 45 kV and 40 mA from 10° to 80° with a Ni-filtered $\text{Cu K}\alpha$ radiation ($\lambda = 1.542 \text{ \AA}$). The Fourier transform infrared (FTIR) spectra were collected from a TENSPPR37 spectrometer (Bruker Co., Germany) at a 4 cm^{-1} resolution from $4,000$ to 500 cm^{-1} via KBr pressed-disk technique. The thermal decomposition behaviors of PCS and as-spun composite nanofibers were recorded by thermogravimetric analysis (TGA) (STA409PC, Netzsch Co., Germany) in nitrogen at $10^\circ\text{C}/\text{min}$ from 25°C to 900°C . The nitrogen adsorption and desorption isotherms were measured at 77 K using a Micromeritics ASAP 2020 M system. The samples were outgassed at 120°C for 3 h before the measurements. The specific surface areas for N_2 were calculated using the Brunauer-Emmet-Teller (BET) model. SiC-NFM-10, SiC-NFM-20, SiC-NFM-30 represented 10 wt.%, 20 wt.%, 30 wt.% of PS concentrations, respectively. And the SiC-NFM-30 was used for the material characterization and analysis.

2.4. Electrochemical Testing

All the electrochemical experiments were conducted on a CHI760D electrochemical workstation. The electrochemical properties were measured by cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS) methods in a classical three electrodes set-up. The working electrode was prepared according to the following steps: The working electrodes were fabricated by pressing the 2.5 mg SiC nanofiber membranes (SiC-NFMs) onto the nickel foam current collector ($1 \times 1 \text{ cm}^2$). In order to ensure adequate contact between

Table 1
Parameters of solution blowing process.

Parameters	Values
PCS concentration	20 wt.%
PS concentration	10 wt.% ~30 wt.%
Solution feeding rate per needle	16 mL/h
Gas pressure	0.12 MPa
Collector distance	60 cm
Diameter of the needle	0.5 mm

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