

# High-performance lithium storage in nitrogen-enriched carbon nanofiber webs derived from polypyrrole



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## ABSTRACT

Nitrogen-doped carbon nanofiber webs (N-CNFWs) are prepared by direct pyrolyzation of polypyrrole (PPy) nanofiber webs at 600 °C. The structure and morphology of N-CNFWs are characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), Raman spectra and elemental analysis. Both the doped N content and the N existing type in carbon, change with the pyrolysis time. As anode material for lithium-ion battery, the N-CNFWs show high capacity and good rate capability. The reversible capacity is up to 668 mAh g<sup>-1</sup> at a current density of 0.1 A g<sup>-1</sup> and 238 mAh g<sup>-1</sup> at 5 A g<sup>-1</sup>, which can be ascribed to the nanofiber structure and high nitrogen content.

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## 1. Introduction

Lithium-ion batteries (LIBs) have risen as important devices for development of green and sustainable energy [1]. Currently, graphite is the commercially available anode material for LIBs owing to low cost, long lifespan and low lithium intercalation potential. However, its low storage capacity (372 mAh g<sup>-1</sup>) and limited rate capability cannot meet the requirements imposed on application of LIBs in high-power devices [2,3]. Therefore, new carbon-based anode materials need to be identified for LIBs.

Recently, nitrogen-doped carbons have been regarded as promising anode candidates for high-performance LIBs. It is well suggested that N-doping process can generate extrinsic defects and form a disordered carbon structure, thus enhancing Li intercalation properties [4–7]. Moreover, N-doping can improve the reactivity and electrical conductivity, leading to enhanced Li<sup>+</sup>-ion storage capacity [8–10]. Nitrogen functionalities may induce many active sites to absorb Li ions, which can enhance the capacity [8,10–13]. Accordingly, the N content and the N type in the N-doped carbon structure play important roles in Li<sup>+</sup> storage performance for LIBs. The carbon with high N doping level exhibits high reversible capacity [14–16]. For the type of N dopant, the pyridinic N is preferable for Li storage whereas the graphitic N is not suitable [10]. However,

the N content in the as-mentioned carbon is usually low, and few researches have paid attention to the effect of N type in the N-doped carbon anode for LIBs.

For the N-doped carbon, synthesis method is crucial for the properties. The common route is post-treatment of carbon together with N-rich precursor (e.g., NH<sub>3</sub>) [14,17–19]. However, this strategy usually shows some disadvantages such as low yield and high cost, which limits its application for large-scale production of N-doped carbon material. In situ doping via carbonization of N-rich conducting polymer is an easy-to-operate method for relatively large-scale production [12,20–23]. Very recently, we successfully prepared porous N-doped carbon nanofiber webs with high N content via carbonization-activation of polypyrrole (PPy) nanofiber webs template by using KOH as activating agent [12]. Such obtained N-doped carbon shows excellent Li storage performance, but the initial coulombic efficiency is low. In this work, we synthesized the high-level N-doped carbon nanofiber webs (N-CNFWs) directly through pyrolyzation of PPy nanofiber webs precursor. We systematically investigated the electrochemical performance of N-CNFWs and focused on the influence of the doped N on the properties of the carbon.

## 2. Experimental

PPy nanofiber webs were synthesized by a modified oxidative template assembly route, as described previously [12,24]. Typically, cetrimonium bromide (CTAB, (C<sub>16</sub>H<sub>33</sub>)N(CH<sub>3</sub>)<sub>3</sub>Br, 7.3 g) was dissolved in diluted HCl solution (120 mL, 1 mol L<sup>-1</sup>) under ice bath.

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Then ammonium persulfate (APS, 13.7 g) was added, and a white reactive template was formed immediately. After being magnetically stirred for 0.5 h, pyrrole monomer (8.3 mL) was added into the as-formed reactive template solution. The reaction was carried out at 0–5 °C for 24 h. A black precipitate (polypyrrole nanofiber webs) was obtained. The precipitate was washed with 1 mol L<sup>-1</sup> HCl solution and deionized water for several times till the filtrate became colorless and neutral. The product was dried overnight at 80 °C in an oven. The as-obtained PPy was heated to 600 °C at a heating rate of 5 °C min<sup>-1</sup> and treated for 0.5–4 h to form nitrogen enriched carbon nanofiber webs. The corresponding obtained products are denoted as N-CNFWs-0.5 h, N-CNFWs-1 h, N-CNFWs-2 h and N-CNFWs-4 h. The yields were about 52%.

The products were characterized by X-ray diffraction with the Rigaku D/MAX-RB diffractometer using filtered Cu K $\alpha$  radiation (XRD, Panalytical X'pert PRO MRD, Holland), scanning electron microscope (Sirion 200, Holland), Raman spectra (Bruker VERTEX 70) and Fourier transform infrared spectrometer (FTIR, Bruker, VERTEX 70). CHNS elemental analysis was performed with an elemental analyzer (Vario Micro cube). X-ray photoelectron spectroscopy (XPS) measurement was performed on a VG MultiLab 2000 system with a monochromatic Al X-ray source (ThermoVG Scientific).

Electrochemical tests were carried out with CR2032 coin cells. The working electrode was fabricated by mixing the active material, acetylene black and PVDF binder with a weight ratio of 80:10:10 in N-methyl-2-pyrrolidone solvent. The resultant slurry was uniformly pasted on Cu foil to form an electrode film, and then dried at 80 °C in vacuum. Metallic lithium foil was used as anode electrode; the electrolyte was 1 mol L<sup>-1</sup> LiPF<sub>6</sub> in a 1:1 mixture solvent of ethylene carbonate and dimethyl carbonate (EC/DMC). A thin sheet of microporous polyethylene (Celgard 2400) served as the separator. The cells were assembled in an argon-filled glove box. Charge/discharge tests were performed between 3 V and 0.01 V with a Land CT2001 battery tester (Wuhan Land Electronic Co. Ltd., China) at 25 °C. Cyclic voltammetry (CV) was measured by an electrochemical workstation (PARSTAT 2273, Princeton Applied Research, US) at a scan rate of 0.1 mV s<sup>-1</sup> between 3 V and 0.01 V.

### 3. Results and discussion

Fig. 1 shows SEM images of PPy nanofiber webs and N-CNFWs and TEM image of N-CNFWs-2 h. It can be seen that the as-obtained PPy shows homogeneous morphology of cross-linked nanofibers with diameter in the range of 70–90 nm (Fig. 1a). The PPy nanofiber webs were carbonized at 600 °C for different times, leading to form

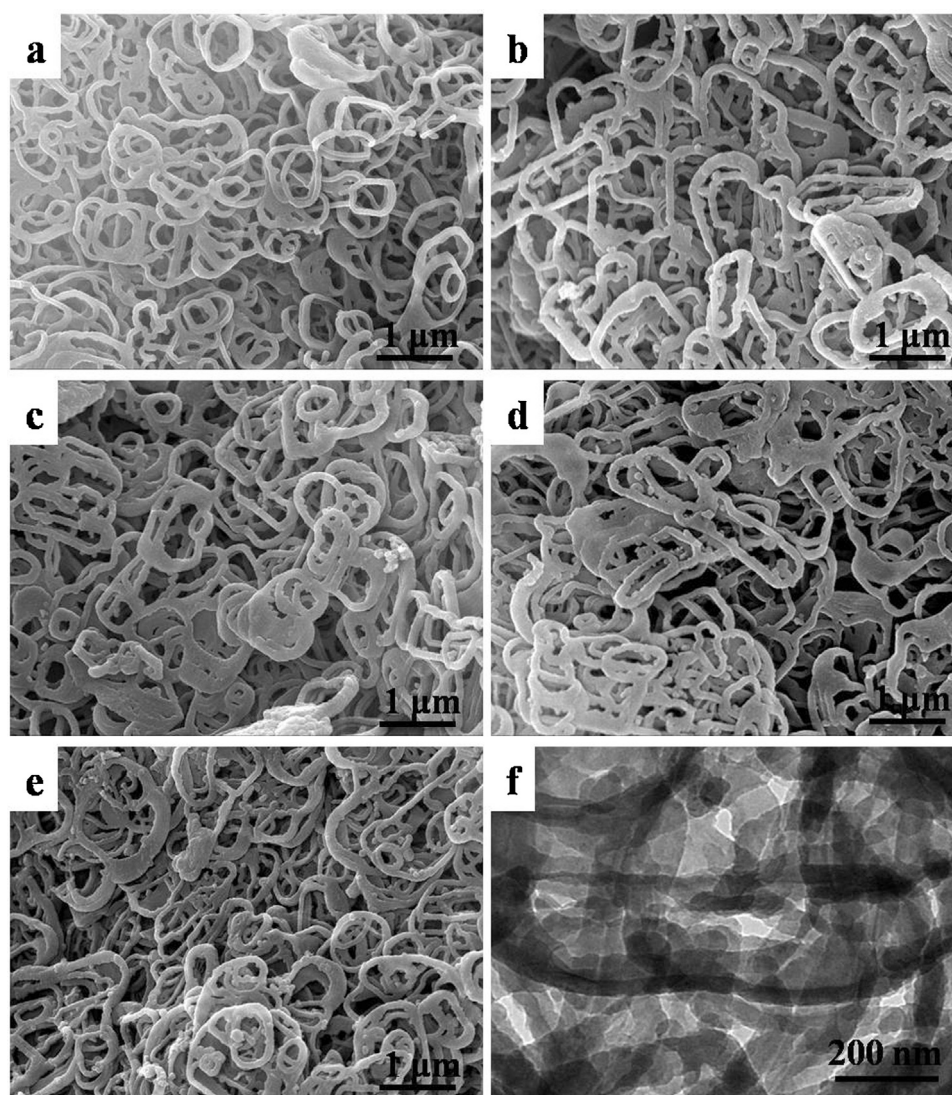


Fig. 1. SEM images of (a) PPy nanofiber webs, (b) N-CNFWs-0.5 h, (c) N-CNFWs-1 h, (d) N-CNFWs-2 h and (e) N-CNFWs-4 h; (f) TEM image of N-CNFWs-2 h.

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