



Short communication

High-performance supercapacitor based on nanocages with nanoporous thin-walled graphitic shells



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ABSTRACT

Thin-walled graphitic nanocages (GNCs) made up from highly nanoporous shells have been prepared by removing nitrogen-doping (N-doping) template. N-doping template is carboxyl groups and nitro-groups, which have formed from oxidation of N-doped structure of GNCs by acid-treatment. After partially removing N-doping template by annealing in vacuum, nanopores are created in the shells of the thin-walled GNCs. Thus, their specific surface area (1100 vs. 700 m² g⁻¹) and specific capacitance (370 vs. 300 F g⁻¹ at current density of 0.1 A g⁻¹) are sharply raised. With current density increasing to 1 A g⁻¹, the capacitance of the GNCs is still high (230 F g⁻¹), indicating such GNCs is a superb electrode material of supercapacitor.

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1. Introduction

As energy storage devices, supercapacitors present an attractive new technology, specifically for high power applications such as hybrid electric vehicles or portable devices [1–10]. The electrical double-layer capacitor is one type of supercapacitors, which stores electric energy by the physical adsorption of electrolyte ions onto a charged electrode by electrostatic attraction [2–6]. Thus, the materials with large specific surface area, such as porous materials, have been preferred to approach high capacitance, traditionally [2–6]. Graphitic nanomaterials, such as carbon nanotubes (CNTs) [11,12], graphenes [6,13] and graphitic nanospheres [14–17], have well-developed crystalline structure leading to their high electric conductivity and oxidation resistance in electrochemical applications [11–17]. However, the well-developed graphitic structure has reduced the defects of such materials leading to low specific surface area (<600 m² g⁻¹) [11–16], which limits specific capacitance of such graphitic materials. To raise specific surface area of graphitic nanomaterials, lots of methods have been carried out. One strategy is trading off graphitic structure by violently chemical treatment, such as treating CNTs in CO₂ or KOH [18,19]. Another strategy is synthesizing carbon nanomaterials made of a few graphitic layers,

such as single walled CNTs [11,12], thin walled carbon nanocages [15–17] and graphenes [6,13]. Additionally, doped carbon nanomaterials have also been prepared for assembling high performance supercapacitors, because doping structure might increase their pseudocapacitance [2,9,10,20].

Here, we demonstrate a novel approach for thin-walled graphitic nanocages (GNCs) made up from highly nanoporous shells prepared by removing N-doped template inserted in the graphitic layers of GNCs. N atoms partially replaced C atoms in the graphitic shells to form doping structure, when the Fe@C core-shell nanoparticles were prepared by thermal pyrolysis and then N-doped GNCs were obtained from removal of their ferrous cores. After removing N-doped template, the specific surface area of thin-walled GNCs was sharply raised and their shells were still well-graphitized. Thus, such GNCs might be a superb electrode material towards high performance supercapacitor applications.

2. Experimental

For preparing graphitic nanocages with high specific surface area, core-shell nanoparticles were prepared by thermal pyrolyzing a gas mixture of ammonia, acetylene and iron pentacarbonyl in nitrogen atmosphere at 750–1050 °C. The typical flow rates were 80 L h⁻¹ for nitrogen, 30 mL min⁻¹ for ammonia and 10 mL min⁻¹ for acetylene, and those gases flowed through the liquid iron

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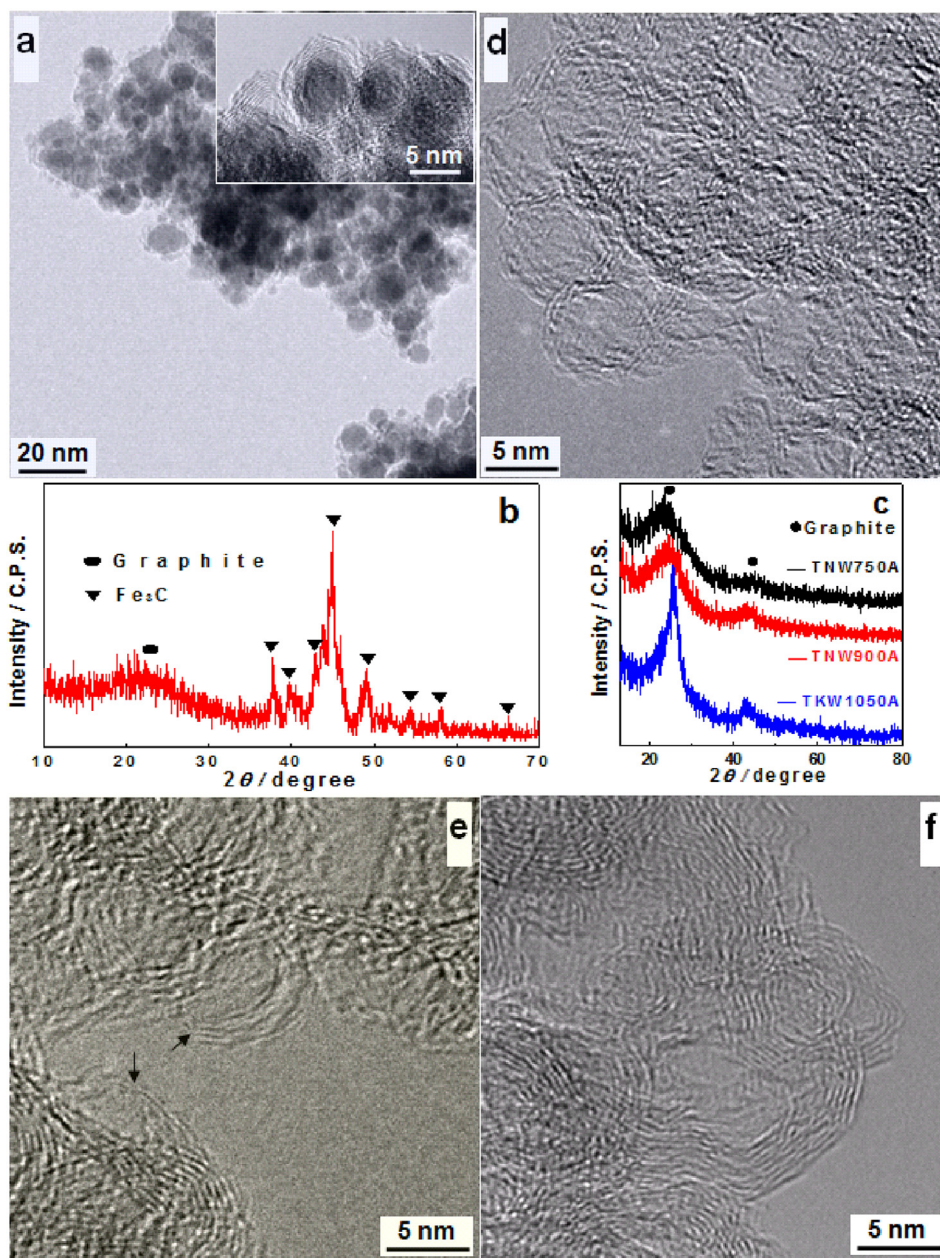


Fig. 1. TEM image (a) and XRD pattern (b) of the precursor (TNW900) prepared by thermal pyrolysis (The precursors have been denoted as TNW750, TNW900 and TKW1050 for distinguishing them by their synthetic temperature). (c) XRD patterns of N-doped GNC samples (TNW750A, TNW900A and TKW1050A) prepared from acid-treating the precursor. TEM images of GNC samples after partially removing N-doping template by 300 °C heat treatment in vacuum: TNW750AH (d), TNW900AH (e) and TKW1050AH (f).

pentacarbonyl to add the chemical into the gas mixture. After thermal pyrolyzing, core-shell nanoparticles were collected at the other end of the quartz tube. To remove the cores, the nanoparticles were stirred in mixed acid ($\text{HCl}:\text{HNO}_3 = 10:1$) at 80 °C for 6 h and then such acid-treated samples were filtered and dried to obtain N-doped graphitic nanocages. For partially removing N-doping template, the samples were heated to 300 °C for 0.5 h in vacuum.

All samples were analyzed by X-ray diffractometer (XRD) (Bruker D8 Advance, Bruker AXS, Germany), high-resolution transmission electron microscopy (HRTEM) (JEOL: JEM-2100F), Fourier transform infrared spectroscopy (FTIR: Nicolet 6700, USA) and X-ray photoelectron spectroscopy (XPS). Nitrogen adsorption/desorption measurement of carbon samples were carried out at 77 K (ASAP 2020, Micromeritics).

Electrochemical experiments were carried out on a CHI 760E electrochemical workstation. The GNC-based electrodes were fabricated by pressing a mixture of 85 wt% active material, 5 wt% acetylene black, and 10 wt% polyvinylidene fluoride (PVDF) onto a piece of nickel foil ($\sim 1.1 \text{ cm}^2$). The electrolytic solution was 6 M KOH. In addition to the working electrode, a saturated calomel electrode (SCE) and a Pt electrode were used as the reference and counter, respectively. The cyclic voltammetry profiles were recorded from the potential of -1 to 0 V versus SCE. Galvanostatic charge-discharge measurements were carried out at current density from 0.1 to 2 A g^{-1} in the voltage range between -1 and 0 V .

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