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Featured Letter

Solvothermal synthesis of antimony phosphate hierarchical microspindles and their capacitive property

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ABSTRACT

$SbPO_4$ hierarchical microspindles are successfully prepared through a facile solvothermal route. The X-ray powder diffraction, scanning electronic microscopy and nitrogen adsorption-desorption techniques are utilized to characterize the obtained $SbPO_4$ materials. It is found out that the as-prepared $SbPO_4$ microspindles are composed of nanoplates with mesoporous features. A self-assembly process is proposed for the formation of the $SbPO_4$ microspindles. Besides, the $SbPO_4$ microspindles is also tested as an electrode for supercapacitor, which demonstrates an enhanced electrochemical property due to their novel nanostructures.

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1. Introduction

The ever-growing demands for renewable energy have encouraged a wide range of investigation to exploit efficient energy storage and conversion devices [1]. Supercapacitors, which are also called electrochemical capacitors, have drawn much attention because of their fast charging capabilities, long-life cycles as well as broad working temperature ranges and therefore have sprung up to be the potential contestants for energy storage and conversion [2].

Supercapacitors can be categorized into an electrical double-layer capacitor (EDLC) and a pseudocapacitor (PC) in terms of the charge storage mechanism. Specially, pseudocapacitors can store charge by redox reactions and accumulate charge at the electrode/-electrolyte interface, and thus can deliver a higher capacitance than EDLCs [3]. Among the commonly reported pseudocapacitive electrode materials, metal oxides with multiple valence states and conducting polymers are widely investigated as pseudocapacitor electrodes [4,5].

Recently, phosphate materials such as transition metal phosphates, for example vanadyl phosphates [6], cobalt phosphates [7] and nickel phosphates [8] are now promising electrode materials for supercapacitors. However, less attention was focused on metalloid-based phosphate as electrode materials. Among the commonly available metalloid phosphates, $SbPO_4$ (antimony phosphate) is a layered phosphate which is composed of layers of tetrahedral PO_4 and polyhedral SbO_4 concatenated via the corners to form an outstretched framework [9]. Moreover, $SbPO_4$ can serve as an electrode for energy storage which originates from the reversible redox transitions between Sb^{3+} and Sb metal states [10]. Meanwhile, the P—O covalent bond of PO_4^{3-} can stabilize Sb^{3+} by the inductive effect of Sb—O—P bond, which makes $SbPO_4$ to be a promising electrode for pseudocapacitors [10–12]. Till date, nanosized $SbPO_4$ materials with different morphologies including particles, nanorods and ribbons, hollow spheres have been prepared [13–15]. However, its application as electrode for supercapacitor are still seldom. Herein, we report a solvothermal method to fabricate $SbPO_4$ hierarchical microspindles assembled from nanoplates, which exhibit an enhanced electrochemical capacitive performance.

2. Experimental procedure

2.1. Synthesis of $SbPO_4$ microspindles

2 mmol of antimony potassium tartrate was dissolved in 60 mL glycerol under continuous stirring. Then 10 mmol of polyphosphoric acid were slowly dropped into the above solution under violent stirring. The resulting colloidal mixture was transferred into a Teflon-lined autoclave and maintained at 180 °C for 6 h. After that, the autoclave was cooled down naturally. The white precipitates were gathered, washed with water and alcohol in sequence and separated by centrifuge, and dried at 60 °C for 12 h in a vacuum oven to obtain $SbPO_4$ hierarchical microspindles ($SbPO_4$-MS). For

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comparison, $SbPO_4$ nanoplates ($SbPO_4$-NP) were also fabricated through a similar hydrothermal route.

2.2. Characterization

The crystal phase and crystallinity were identified by X-ray diffraction (XRD, D/Max-2550) with monochromatized CuKα radiation. The morphology and microstructure were inspected by scanning electron microscope (SEM, FEI SIRION-100). The Brunauer-Emmett-Teller (BET) surface area was estimated from the adsorption data obtained with the nitrogen sorption analysis (Quantachrome NOVA 2000e).

2.3. Electrochemical measurements

Electrochemical property of the supercapacitor was tested by cyclic voltammetry (CV) and galvanostatic charge/discharge (GCD) techniques at an electrochemical station (CHI660E). The

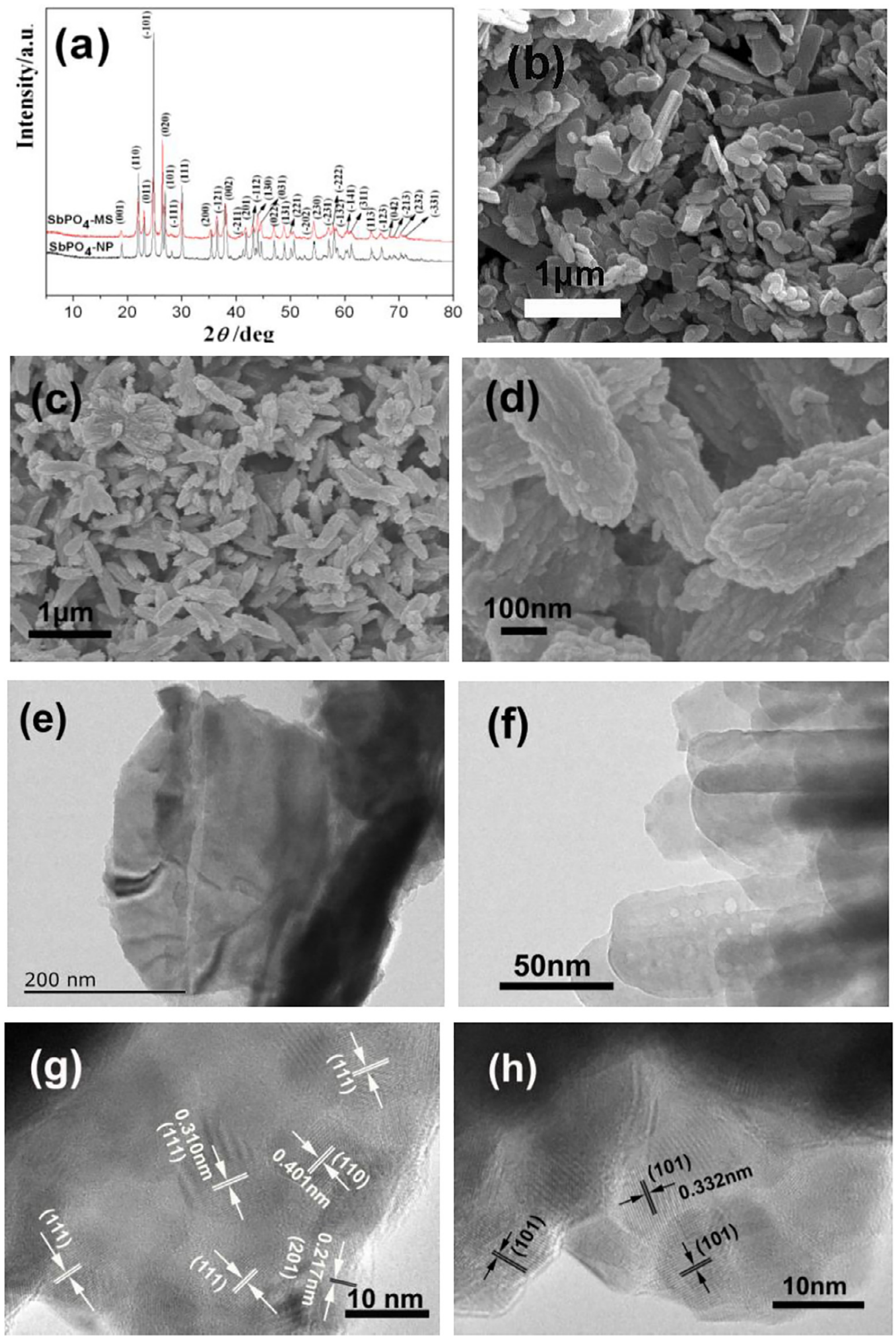

Fig. 1. (a) XRD patterns and SEM images of (b) $SbPO_4$-NP and (c and d) $SbPO_4$-MS; TEM and HRTEM images of (e and g) $SbPO_4$-NP and (f and h) $SbPO_4$-MS.

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