Journal of Alloys and Compounds 737 (2018) 623–629

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Microwave-assisted synthesis method for rapid synthesis of tin selenide electrode material for supercapacitors

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ARTICLE INFO

Article history:
Received 16 September 2017
Received in revised form
28 November 2017
Accepted 4 December 2017
Available online 6 December 2017

Keywords:
Tin selenide
Microwave-assisted synthesis
Supercapacitor

ABSTRACT

As an important binary IV-VI semiconductor compound, tin selenide (SnSe) has been investigated intensively for a wide range of applications in energy storage and photovoltaic devices, due to its unique electronic and optoelectronic properties. In this work, we successfully synthesized SnSe powders by a simple, rapid and high-yield method called microwave-assisted synthesis for the first time and also measured their electrochemical performances. By rationally controlling the microwave heating time, we found that the 15-min reacted sample exhibited the most outstanding specific capacitance and rate capability (214.3 F/g at 1 A/g and 182.8 F/g at 20 A/g), and excellent cycling stability. The microwave-assisted synthesis method is efficient and rapid for preparing SnSe electrode materials.

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1. Introduction

Energy consumption and environmental pollution have been the focus of attention for mankind today. With the development in clean and sustainable energy from solar cells and wind turbines, efficient energy storage devices are needed to harvest and store the electricity derived from the renewable and sustainable energy [1–4]. Most of all, exploring new, highly efficient, and mass-produced materials for energy storage devices is one of the hot spots of current researches.

Tin selenide (SnSe) belongs to one class of typical layered metal chalcogenides [5], and it possesses an orthorhombic structure (space group: *Pnma*) with an indirect band gap at 0.9 eV and a direct band gap at 1.30 eV [6], hence, exhibiting a unique electronic structure and various excellent physical properties. Meanwhile, SnSe has attracted interest as a stable and simple binary compound with nontoxic and earth-abundant elements [6]. Given all this, it is regarded as a very potential candidate for solar cells [6–8] and thermoelectric materials [9,10]. The synthesis of other tin chalcogenides including SnTe and SnS has been reported [11–14], nevertheless, the synthesis of SnSe nanocrystals for electrode material is not very successful due to the appearance of $SnSe_2$ [15] besides SnSe in the final product [6,16].

Until now, numerous methods for preparing SnSe have been reported. Butt et al. [17] used chemical vapor deposition technique to synthesize SnSe nanoparticles. Ramasamy et al. [18] prepared SnSe by a solvothermal synthesis method. Fernandes et al. [19] utilized a magnetron sputtering method to synthesize SnSe. All the conventional methods mentioned above for preparing SnSe need complex crafts, long preparation time as well as high energy. Compared with conventional heating methods, a microwave-assisted synthesis method can accomplish internal volumetric heating by efficiently and rapidly transferring microwave energy into the reaction mixture [20]. Thus, it is able to nucleate instantaneously, and the process is very fast and saves energy. Besides, it possesses many other fascinating merits including simple raw materials, uniform heating, high-yield, and better selectivity [21–23]. This method has been widely used for synthesizing of various materials. Voiry et al. [24] obtained high-yield graphene via microwave reduction of graphene oxide. Harpeness and Gedanken [25] synthesized nanostructured Bi_2Se_3 by a microwave-assisted synthesis method. Jiang et al. [26] reported preparation of Bi_2Te_3 plate-like crystals with homogeneous hexagonal morphology by a microwave-assisted synthesis method. Our research group recently also successfully fabricated Bi_2Te_3 nanostructures with various morphologies (microrods, nanoplates, and nanoflowers) [27,28]and MoS_2 nanosheets [29] by a microwave-assisted synthesis method.

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https://doi.org/10.1016/j.jallcom.2017.12.037

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Electrochemical capacitor, also called supercapacitor offers manifold excellent characteristics including high power density, fast charging and discharging capability, superior cycle lifetime, high reliability and security [30,31], which makes it to be one of the hottest members among various energy storage devices [32,33]. As known to all, the electrode material is the critical factor for the eventual properties of a device. Many excellent works on electrode materials have been reported, such as MoO_3 [34], $CoCO_3$ [35], MoS_2 [36], polypyrrole(PPy) [32], PPy/V_2O_5 [37], PPy/MoO3 [38], PPy/MoS_2 [33]and so on. To the best of our knowledge, only hydrothermal synthesis method has been used for preparing SnSe electrode material for supercapacitors [39] and the synthesis of SnSe by the microwave-assisted synthesis method has never been reported until now.

In this work, we used the microwave-assisted synthesis method to prepare SnSe electrode material for supercapacitors. We successfully synthesized SnSe particles using $SnCl_2$ as the tin source and trioctylphosphine (TOP)-Se as the selenide source with the help of borane-*tert*-butylamine complex (BTBC) and 1,3-dimethyl-3,4,5,6-terahydro- 2(1H)-pyrimidinone (DMPU) according to ref. [39] which utilized a hydrothermal synthesis method for preparing SnSe. The role of each reagent is as follows: (1) DMPU acts as a solvent and a weak oxidizing agent. (2) BTBC plays the role of a reducing agent and also a morphology inhibiting reagent. (3) TOP is a solvent for Se powder and a morphology inhibiting reagent that binds with BTBC to realize selective growth.

For comparison, we prepared three different samples by microwave heating for 5, 10 and 15 min, respectively. And the specific capacitance, rate capabilities and cycling stabilities of the samples were analyzed.

2. Experimental

2.1. Raw materials

Anhydrous $SnCl_2$ and Se powder were purchased from Sinopharm Chemical Reagent Co., Ltd. borane-*tert*-butylamine complex (BTBC), 1,3-dimethyl-3,4,5,6-terahydro-2(1H)-pyrimidinone (DMPU) and trioctylphosphine (TOP) were obtained from J&K Scientific. All the reagents were used directly without further purification.

2.2. Preparation of tin source

0.12 g of $SnCl_2$ and 1.5 g of BTBC were dissolved in 30 mL DMPU under mild stirring for 30 min at room temperature.

2.3. Preparation of selenide source (TOP-Se)

0.632 g of Se powder was added into 20 mL TOP and stirred for 30 min at room temperature.

2.4. Synthesis of SnSe particles

1.5 mL of TOP-Se was added to the tin source and stirred for 20 min at room temperature, and then the solution was poured into a microwave reactor. The reactor was placed in the household microwave oven (Galanz, P70F23P-G5(SO)). The microwave-assisted reaction was lasted for 5, 10 and 15 min, respectively, with a medium-low fire and then naturally cooled to room temperature, and the corresponding samples were called S-1, S-2 and S-3, respectively.

The products were collected and washed with heptane and absolute ethanol in sequence for several times, then separated by centrifugation at 4000 rpm for 5 min and finally dried in vacuum at 333 K for 8 h for further characterization and analysis.

3. Characterization

The powders were examined by X-ray diffraction (XRD, Bruker D8 Advance) using Cu Kα (λ = 1.5406 Å) radiation. X-ray photoelectron spectroscopy (XPS) analyses were carried out on an X-ray photoelectron spectrometer (XPS, ESCALAB 250Xi). The morphology and microstructure of the samples were observed by field-emission scanning electron microscopy (FESEM) (Nova NanoSEM 450) and high resolution transmission electron microscopy (HRTEM) (JEM-2100F).

3.1. Electrochemical measurements

In a three-electrode system, the as-prepared SnSe particles coated on nickel foam, a platinum sheet, and a saturated calomel electrode (SCE) were used as the working electrode, counter electrode, and reference electrode, respectively. The working electrode was composed of active SnSe particles, conductive material (acetylene black, 20 wt %) and binder [poly(tetrafluoroethylene) (PTFE), 10 wt %]. The mixture was first coated onto the surface of a piece of nickel foam sheet (2 cm × 1 cm) and then dried at 50 °C under vacuum for 12 h.

The electrochemical performance measurements were carried out at room temperature by the electrochemical workstation (CHI 627C). The cyclic voltammogram curves (CVs) were measured at different scan rates (5, 10, 20, 50 and 100 mV/s). The galvanostatic charge-discharge (GCD) curves were tested at various current densities of 1, 2, 5, 10 and 20 A/g, respectively. The specific capacitances (C_s) of the electrode materials were calculated from GCD curves via the following equation: $C_s = I \times \Delta t/(\Delta V \times m)$, where I, Δt, ΔV and m denote discharge current (A), the discharge time (s), potential window (V) and practical active mass (g), respectively.

Electrochemical impedance spectroscopy (EIS) tests were carried out in the frequency range from 100 kHz to 0.01 Hz at open circuit potential using Bio-logic VMP3.

4. Results and discussion

The reaction scheme for the synthesis of SnSe is shown in Fig. 1. Fig. 2 shows the XRD patterns of the samples S-1, S-2 and S-3. All the characteristic peaks except that marked by "*" in the XRD patterns of the S-1, S-2 and S-3 can be well indexed to the orthorhombic SnSe (JCPDS No. 89-0232). Meanwhile, impurity phase Sn (JCPDS No. 04-0673) is detected in the S-1 and S-2 (the intensity of the Sn diffraction peaks marked by "*" in the S-1 is stronger than that in the S-2) but not in the S-3. Thus, the reaction is more completely as the reaction time prolongs, and pure crystalline SnSe is successfully synthesized when the microwave heating time is 15 min.

Moreover, the three samples were further characterized via XPS analysis (Fig. 3). The XPS spectra confirm the presence of the Sn and Se elements for all the three samples. As shown in Fig. 3, the Se 3d peaks of the S-1(d), S-2(e), S-3(f) are located at 53.3 eV(Se $3d_{3/2}$)and 54.3 eV (Se $3d_{5/2}$); however, the Sn 3d peaks of the S-1 and S-2 are much different from that of the S-3. The Sn $3d_{3/2}$ peak of the S-1(a) and S-2(b) are resolved into two individual peaks by using a curve-fitting procedure (marked as 487.0 and 485.6 eV for the S-1 and 486.6 and 485.1 eV for the S-2) due to the co-existence of SnSe and Sn; whereas the Sn $3d_{3/2}$ peak (494.8 eV) of the S-3(c) is consistent with the previous reports of pure SnSe [40,41]. This further confirms that the S-1 and S-2 samples contain impurity Sn besides SnSe and that the S-3 is pure SnSe. The formation mechanism of the SnSe can be described by the chemical reactions as follows:

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