



Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Preparation and characterization of SrLi₂Ti₆O₁₄@C/Ag as lithium storage anode material for rechargeable batteries

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ARTICLE INFO

Keywords:

SrLi₂Ti₆O₁₄
C/Ag coating
Anode material
Lithium rechargeable batteries

ABSTRACT

In this work, silver and carbon co-coated SrLi₂Ti₆O₁₄ is synthesized by using a solid-state assisted solution method, with glucose as carbon source and silver nitrate as Ag source. The structural and morphological properties of as-prepared samples are characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), which confirm that C/Ag composite layer is uniformly coated on the surface of SrLi₂Ti₆O₁₄. Electrochemical measurements like galvanostatic charge/discharge tests, rate performance, cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) analysis are also undertaken to evaluate and compare the lithium storage capability of SrLi₂Ti₆O₁₄ before and after coating. According to the results, SrLi₂Ti₆O₁₄@C/Ag presents enhanced electrochemical capability compared with bare material. It can be found that bare SrLi₂Ti₆O₁₄ only delivers the reversible capacity of 140.32 mA h g⁻¹ with capacity retention of 90.7% at 100 mA g⁻¹ after 200 cycles. In contrast, SrLi₂Ti₆O₁₄@C/Ag presents the reversible capacity of 151.20 mA h g⁻¹ with only 6.7% capacity loss after 200 cycles. The improvement is owing to the increase of electronic conductivity and the decrease in the redox polarization after coating. In order to further investigate the structural stability of SrLi₂Ti₆O₁₄@C/Ag, *in-situ* XRD was performed as well. All the results prove that the C/Ag co-coating has positive effect on the electrochemical performance of SrLi₂Ti₆O₁₄.

1. Introduction

Rechargeable lithium-ion batteries (LIBs) have been widely used in a variety of portable electronic devices because of their various superiorities, such as high voltage, high energy density, stable cycling and environmental-friendly [1]. Furthermore, it proved that LIBs can be used as power battery for high power devices like electric vehicles and hybrid electric vehicles [2,3]. In the case of urgent demand for high performance LIBs, finding a stable lithium storage material with high capacity and high power is increasingly significant for the core work of improvement of electrochemical performance [4]. Recent years, many researchers have focused on the spinel Li₄Ti₅O₁₂ (LTO) anode, a promising titanate anode material applying in both electric vehicles and large stationary power supplies [5]. As a zero strain host material [6], LTO displays small volume change, exceptional high rate performance, excellent cycling stability, the relatively steady operating voltage at about 1.55 V (*vs.* Li/Li⁺) and the theoretical capacity of 175 mA h g⁻¹ during the charge/discharge process [7–9].

Recently, as a new kind of titanate, SrLi₂Ti₆O₁₄ triggered the

attention of many researchers and has been widely investigated. Since a new phase composed of Sr, Li and Ti was discovered from the SrO-TiO₂-LiBO₂ system by Koseva in 1991 [10], different types of titanate materials have drawn considerable attention as high voltage anode materials. The single crystal of SrLi₂Ti₆O₁₄ has been grown by the flux method and its structure was determined in 2002 [11]. I. Koseva reported that MLi₂Ti₆O₁₄ (M=Ba, Sr, Pb) is a new family of isostructural titanates, which can be one of the promising materials to replace graphitic anodes in LIBs [12]. According to the work of D. Dambournet, SrLi₂Ti₆O₁₄ showed superior capacity and rate capability to BaLi₂Ti₆O₁₄ and Na₂Li₂Ti₆O₁₄ [13]. Due to the outstanding cycling stability and rate capability of SrLi₂Ti₆O₁₄ that showed by D. Dambournet, a 2.7-V full cell has been fabricated by combining a SrLi₂Ti₆O₁₄ anode with a 4-V spinel cathode of LiMn₂O₄, which has better performance than the 2.5-V LiMn₂O₄/Li₄Ti₅O₁₂ system [14].

Although the electrochemical performance of SrLi₂Ti₆O₁₄ is quite well, there are still some disadvantages need to be improved, especially for its preparation routes. To make it be a welcomed commercial anode, many efforts have also been paid to investigate the synthesis

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<http://dx.doi.org/10.1016/j.ceramint.2017.03.013>

Received 18 February 2017; Received in revised form 28 February 2017; Accepted 2 March 2017
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reactions of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ for optimizing the preparation methods [15,16]. Recently, Li reported a simple electrospinning route for synthesizing ultra-long one-dimensional $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ nanowires constructed from attached single-crystalline nanoparticles. It is found that the electrochemical performance of nanowires was greatly improved compared to bulks [17]. This work demonstrates that the electrochemical performance of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ still has room to be improved. As well-known, coating is an effective route to enhance the lithium storage capability of titanates [18,19]. For instances, G.J. Zhang prepared carbon-coated spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode material by heat-treating the mixture of TiO_2 (anatase), LiCO_3 and sugar. According to the results of electrochemical tests, it is known that the carbon-coated $\text{Li}_4\text{Ti}_5\text{O}_{12}$ delivers a larger lithium ion diffusion coefficient, better rate capability and reversibility compared with bare $\text{Li}_4\text{Ti}_5\text{O}_{12}$ [18]. To our knowledge, there is no report about the preparation of silver and carbon co-coated $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ as anode material. In this work, $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}/\text{Ag}$ was synthesized by a solid state assisted solution method, and its structure and electrochemical performance were investigated by various measurements, including *in-situ* XRD. The result shows that the improved cyclability and rate performance of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ can be achieved after C/Ag coating.

2. Experimental

2.1. Synthesis

$\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ was synthesized by a simple solid-state reaction. The precursor is obtained from stoichiometric mixture of SrCO_3 (AR, Aladdin), Li_2CO_3 (AR, Aladdin), TiO_2 (99.5%, Aladdin) by 10 h ball milling process. The resulting powder was annealed in a muffle furnace at 600 °C for 4 h and then at 950 °C for another 10 h.

$\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}/\text{Ag}$ was prepared as follows. Firstly, $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ and $\text{C}_6\text{H}_{12}\text{O}_6$ at a mass ratio of 2:1 were mixed in 20 ml distilled water,

stirred for 1 h at room temperature and then dried at 100 °C for 24 h in a vacuum oven. Next, the sample was annealed at 600 °C for 4 h in a muffle furnace under Ar atmosphere to get $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}$. Secondly, the resulting $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}$ powder was suspended in a silver nitrate solution under stirring. After that, the solution of sodium hydroxide was added drop by drop to deposit Ag^+ . Finally, the suspended powder was filtered from the solution and dried for 6 h at 100 °C in a vacuum oven. Here, the mass ratio of AgNO_3 and $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}$ was 5:95; and the molar ratio between AgNO_3 and NaOH was 1:1.

2.2. Characterization and electrochemical measurement

Powder X-ray diffraction patterns were collected on a Bruker D8 Focus diffractometer with Cu K α radiation. *In-situ* XRD measurement was also carried out on it. The surface morphology and microstructure of as-prepared phases were observed by Hitachi SU70 scanning electron microscopy (SEM) equipped with Oxford energy-dispersive X-ray spectroscopy (EDS).

For electrochemical investigation, CR2032 coin-type cells were assembled with round disks as working electrodes consisted of active materials, carbon black and polyvinyl difluoride binder at a mass ratio of 8:1:1. The active loading mass of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ and $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}/\text{Ag}$ in the working electrodes is about 2.0 mg. On LANHE CT2001A battery testing system, galvanostatic charge/discharge experiments and rate performance were measured in a working voltage range from 0.5 to 2.0 V. And on CHI 1000B electrochemical workstation, cyclic voltammetry tests and electrochemical impedance spectroscopy analysis were conducted.

3. Result and discussion

Fig. 1 shows the XRD patterns and structures of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ and $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}/\text{Ag}$. It can be found that all observed diffraction peaks

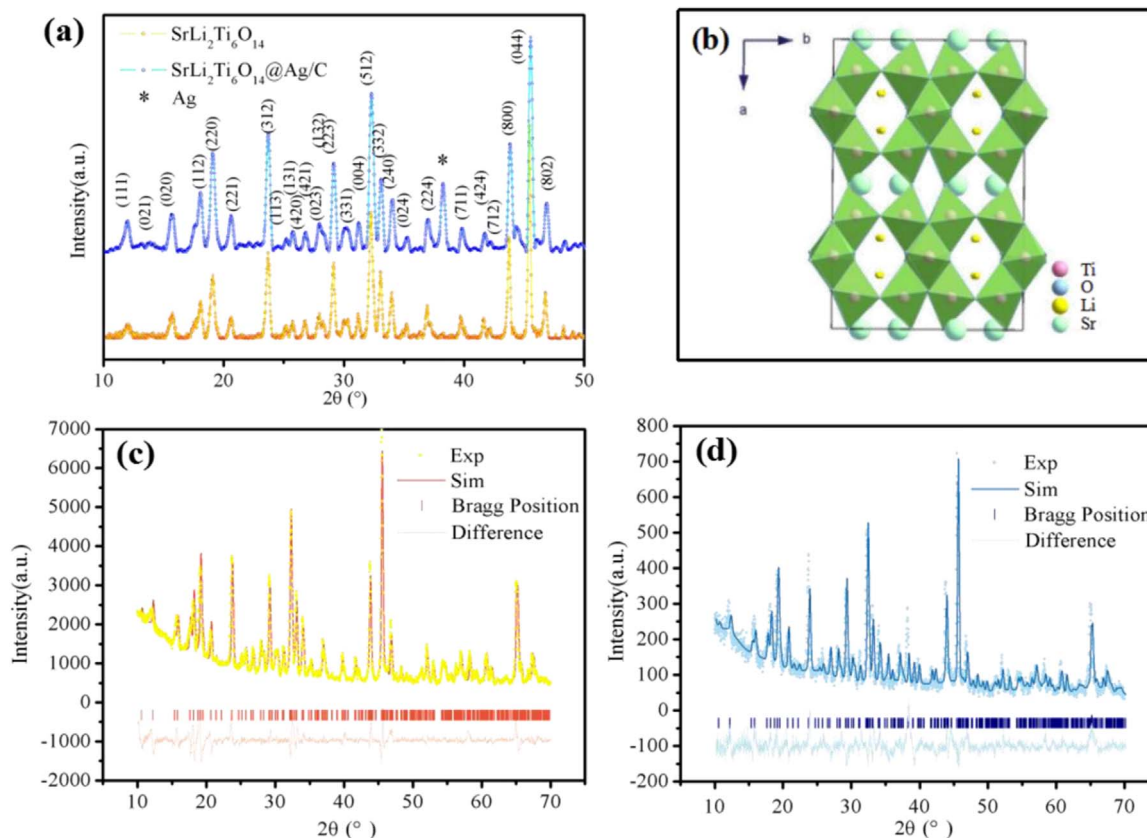


Fig. 1. (a) XRD patterns of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ and $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}/\text{Ag}$, (b) the crystal structure of $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$, Rietveld refinement profiles of (c) $\text{SrLi}_2\text{Ti}_6\text{O}_{14}$ and (d) $\text{SrLi}_2\text{Ti}_6\text{O}_{14}@\text{C}/\text{Ag}$.

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