



Electrochemical performance of silver vanadate/silver nanowire composite for lithium-ion batteries



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ABSTRACT

Silver vanadate ($\text{Ag}_4\text{V}_2\text{O}_7$) nanowires are synthesized through a simple and facile cation exchange method, and the silver vanadate/silver ($\text{Ag}/\text{Ag}_{0.68}\text{V}_2\text{O}_5$) nanowires are obtained through calcination of $\text{Ag}_4\text{V}_2\text{O}_7$ nanowires at 250–500 °C. By varying the reaction time, the progression of the cation exchange process is captured, and tunable morphologies, crystal structures and chemical components (Ag, V, Na and O) of the samples are achieved and analyzed using scanning electron microscope, transmission electron microscope, X-ray diffraction and X-ray photoelectron spectroscopy, respectively. Also, as an electrode of lithium-ion battery, the $\text{Ag}/\text{Ag}_{0.68}\text{V}_2\text{O}_5$ nanowires exhibit specific discharge capacities of 179.5 mAh g^{-1} in the first cycle and 152.6 mAh g^{-1} at the 150th cycle at a current density of 30 mA g^{-1} . Good lithium-ion storage performance of the electrode mainly due to its nanowire structure directly grown on titanium foil and good electron transportation caused by the high conductivity of the silver nanoparticles in the nanowires.

1. Introduction

Lithium-ion primary and rechargeable batteries represent one of the most important developments in energy storage and conversion in the past century [1], due to their high energy density, long cycling life, no/little memory effect, low self-discharge effect and benign environmental impact [2,3]. Owing to their unique electrical properties, abundance resource and low cost [4,5], vanadates have recently been extensively studied in the field of lithium-ion batteries (LIBs). There have been a lot of applications of vanadates as electrode materials of LIBs [6–10]. For example, $\text{NH}_4\text{V}_4\text{O}_{10}$ nanowires on Ti foil can deliver a higher discharge capacity of 330.5 mAh g^{-1} after 100 cycles at 0.8–4.0 V at 50 mA g^{-1} [6], and LiV_3O_8 nanosheets show a discharge capacity of 262 mAh g^{-1} after 100 cycles at 100 mA g^{-1} in a voltage range of 1.5–4.0 V [11].

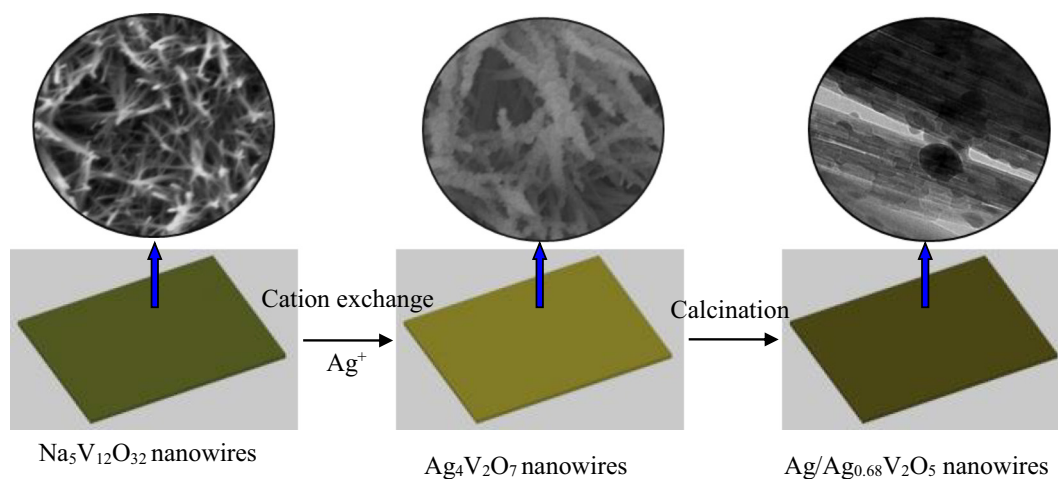
Many vanadates (e.g. sodium vanadate) have layered structures, and the cations are located between these layers. Since the cations can run out from the layers during the charge process in a LIB, it is possible to utilize them for cation exchange with other ions, which can largely expand the range of nanoscale materials with diverse composition, structure, and shape [12,13]. Researchers have successfully employed

the ion exchange method for synthesizing silver vanadium oxides using vanadium oxide xerogels [14–16], while such reactions often take a very long time to complete from several weeks to months [16]. In nanomaterials, the reaction barrier for nucleation is reduced, where high curvature surfaces and low co-ordination facets could serve as high-energy sites ideal for nucleation [17]. In fact, cation-exchange reactions in nanocrystals are up to 100 times faster than those in bulk materials [18].

Silver vanadate, a member of transition metal vanadate, has been extensively studied by researchers for its excellent electrochemical properties and organic pollution degradation [19–22]. As a layered compound consisting of deformed VO_6 octahedra sharing edges and corners [23], $\text{Ag}_{0.68}\text{V}_2\text{O}_5$ was first proposed as a potential electrode material in 1992 [24,25]. To our knowledge, many works of vanadates are reported, as V element shows a range of oxidation states from +2 to +5, having the potential to offer high capacities for lithium-ion batteries [26–28]. Silver loading on the surface of electrode material can bring a series of benefits. Takeuchi et al. [29,30] exhibited that the silver particles could increase the high current capability of the electrode. $\text{Ag}/\beta\text{-AgVO}_3$ hybrid nanorods prepared by Pan et al. [31] showed high discharge capacity, excellent rate performance, and improved

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Scheme 1. Scheme of the fabrication route to Ag/Ag_{0.68}V₂O₅ nanowires.

cycle stability. Peony-like Ag/Ag_{0.68}V₂O₅ presented superior electrochemical properties as both anode and cathode materials for rechargeable lithium-ion batteries [32]. In this work, Ag₄V₂O₇ nanowires are prepared through an efficient and simple cation-exchange method using Na₅V₁₂O₃₂ nanowires as precursors. Scheme 1 illustrates the formation procedure of Ag/Ag_{0.68}V₂O₅ nanowires. Further, Ag/Ag_{0.68}V₂O₅ nanowires are obtained by annealing Ag₄V₂O₇ nanowires in N₂. The morphology, crystal structure, and elemental component of the products are evaluated and the lithium storage properties of the nanowires are investigated and presented.

2. Materials and methods

2.1. Preparation of Na₅V₁₂O₃₂ precursor

In a typical synthesis, a mixed solution of the molar ratio of NH₄VO₃ (0.28 mol L⁻¹): NaCl: H₂C₂O₄·2H₂O : hexamethylene tetramine (HMTA) is 1:6:2:0.2 added into a 50 mL Teflon-lined stainless steel autoclave liners and heated to 150 °C for 1 h to synthesize Na₅V₁₂O₃₂ nanowires arrays on Ti foil [33]. Subsequently, the samples are washed with distilled water for three times and dried at 100 °C for 12 h in air.

2.2. Preparation of Ag/Ag_{0.68}V₂O₅

The Na₅V₁₂O₃₂ nanowires on Ti foil are immersed in 25 ml of 0.3 M AgNO₃ aqueous solution, placed in darkness for 12 h at room temperature. After reaction, the products are washed with distilled water, and dried at 60 °C. Finally, Ag/Ag_{0.68}V₂O₅ on Ti foil is annealed at 250, 300, 400 and 500 °C for 2 h with a heating rate of 1 °C/min in a tube furnace filled with nitrogen atmosphere.

2.3. Characterization

The samples are characterized by Field-emission scanning electron microscope (FE-SEM, Hitachi S-4800 at 15 kV), Scanning electron microscope (SEM, JEOL JSM-6360LV at 20 kV), and Field-emission transmission electron microscope (TEM) JEOL-2100F at 200 kV, with a high-angle annular dark-field (HAADF) detector for imaging in the scanning TEM (STEM) mode. Bruker D8 X-ray diffractometer (XRD) and X-ray photoelectron spectrometer (XPS, KAlpha 1063, Thermo Fisher Scientific, UK) are used to analyze the crystalline structure and chemical compositions of the samples. Fourier transform infrared spectroscopy (FTIR) of the sample is measured on a Fourier transform spectrometer (BRUCKER Tensor 27) at 25 °C in the range of 400–2000 cm⁻¹.

2.4. Electrochemical measurements

Electrochemical performances are tested using coin cells (CR2016). The samples calcined at 250, 300, 400 and 500 °C are directly used as the working electrode with Li metal as counter electrode and 1 M LiPF₆ (EC: DMC: EMC = 1:1:1, in volume) as electrolyte for the half cells, which are assembled in an argon-filled glove box. Cyclic voltammetry (CV) studies are carried out with a scanning rate of 0.3 mV s⁻¹ in the voltage range of 2.0–4.0 V on an electrochemical workstation (Corrtest Instruments, model CS120). The galvanostatic discharge/charge tests are conducted under various current densities with a Neware battery tester (CT 3008) in the voltage range of 2.0–4.0 V. All the tests were measured at 25 °C.

3. Results and discussion

Fig. 1(a) presents XRD patterns of samples obtained via cation exchange of the Na₅V₁₂O₃₂ nanowires in an AgNO₃ solution at room temperature for different times. After the treatment for 2 h, diffraction peaks of Ag₄V₂O₇ phase emerge, suggesting that the Ag⁺ ions have exchanged into the Na₅V₁₂O₃₂ phase. The diffraction peaks of Na₅V₁₂O₃₂ phase gradually weaken as the reaction time is prolonged, meanwhile the diffraction peaks of the Ag₄V₂O₇ phase gradually intensify. When the treatment time prolongs to 12 h, the Na₅V₁₂O₃₂ phase have completely transformed to the Ag₄V₂O₇ phase. The XRD patterns of the Ag₄V₂O₇ calcined at different temperatures are shown in Fig. 1(b). The as-prepared sample has low crystallinity, and the sample crystallinity gradually increases with increasing calcination temperatures. While, when the heat-treatment temperature reaches to 250 °C, the Ag₄V₂O₇ phase has transformed to the monoclinic phase Ag_{0.68}V₂O₅ (JCPDS No. 74-2407). All calcined samples contain reflections from cubic phase Ag metal (marked with ▼), with a space group of *Fm-3m* (JCPDS No. 04-0783).

Fourier transform infrared spectroscopy (FTIR) spectra of Na₅V₁₂O₃₂ nanowires precursors, cation exchanged with Ag⁺ with different times are displayed in Fig. 2. All spectra have a strong absorption at 540 cm⁻¹, which is assigned to the symmetric stretching vibration of V–O–V [34]. As the cation exchange time prolongs, a peak located at 748 cm⁻¹, which corresponds to the asymmetric stretching vibration of V–O–V, is weakened. While, new peaks ascribing to vibrations of O–(V)₃– appear at 820 cm⁻¹ [35], which may be due to the structural arrangement accompanying the substitution of Na ions. For pure Na₅V₁₂O₃₂, the characteristic absorption peak at 950 cm⁻¹ is the stretching vibration mode of the V=O band [36]. As the exchange time is extended, the absorption peak of the V=O band shifts to the lower wave numbers, indicating a lengthening of the V=O bond, possibly

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