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Facile synthesis of Nb_2O_5 nanobelts assembled from nanorods and their applications in lithium ion batteries

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ABSTRACT

Hierarchical 1D Nb_2O_5 nanobelts are successfully synthesized *via* a facile solvothermal method and following thermal treatment. The as-formed Nb_2O_5 nanobelts are characterized by XRD, FESEM, TEM, and BET, and the results indicate that they possess pseudohexagonal structure and are composed of ultranarrow nanorods with an average diameter of *ca.* 15 nm. When used as anodic materials for lithium ion batteries, the obtained Nb_2O_5 nanobelts can deliver initial discharge capacities of 209.3 mAh g^{-1} at the current density of 0.5 C. In addition, the Nb_2O_5 nanobelts exhibit a reversible capacity of 95.8 mAh g^{-1} after 200 cycles at relatively high current density of 5 C. The good electrochemical performance of the Nb_2O_5 nanobelts may be ascribed to their good monodispersity, high specific surface areas, and narrow rod-like building blocks. The Nb_2O_5 nanobelts can be developed as promising anodes for high-rate 2 V LIBs with good safety.

1. Introduction

Lithium ion batteries (LIBs), as a fast-developing technology in electric energy storage, have made considerable contribution to several fields [1]. Most recently, with the development of micro/nanoelectronic devices, tremendous attention has been paid to the exploration of small, safe, and powerful LIBs with low operating voltage (2 V *vs.* Li^+/Li) [2]. Nb_2O_5 is considered as an appealing anode material for 2 V LIBs owing to its high valence state and good structural stability [3,4]. Moreover, similar to $Li_4Ti_5O_{12}$, Nb_2O_5 possesses excellent safety advantages due to its appropriate operational voltage plateau (1.0–2.0 V *vs.* Li^+/Li), which can prevent the growth of lithium dendrites after long charge-discharge process and suppress the formation of SEI layers [5–7]. More importantly, compared with $Li_4Ti_5O_{12}$ (175 mAh g^{-1}), Nb_2O_5 has a higher theoretical capacity of 200 mAh g^{-1} [2]. So, Nb_2O_5 has attracted increasing attention in the fields of LIBs, especially in 2 V LIBs [8,9].

Recently, it has been demonstrated that the constructing of nanostructured anode materials can be used to reduce the diffusion length of Li^+ ion, leading to improved electrochemical performance [10]. Furthermore, it is known that the electrochemical properties of nanoscale electrodes are closed related to their sizes and morphologies [11–13]. Thus, up to date, several Nb_2O_5 nanomaterials with various morphologies, including nanorods, hollow nanospheres, and nanosheets, have been successfully prepared to enhance their electrochemical properties [14–16]. Most especially, 1D nanostructured electrodes have achieved great interest because of their high surface areas, enhanced kinetic, and improved electrochemical properties [17–19]. In these regards, it is imperative to probe novel and effective methods of preparing 1D Nb_2O_5 nanomaterials with special morphologies and excellent properties.

Herein, we report a novel and simple solvothermal route and following thermal treatment to synthesize 1D Nb_2O_5 nanobelts. The Nb_2O_5 nanobelts are assembled by "oriented attachment" of ultranarrow nanorods. The Li-ion storage performance of the as-formed Nb_2O_5 nanobelts is researched, and the results indicate that the Nb_2O_5 electrode possesses good electrochemical properties, including high reversible capacity and good rate performance.

2. Material and methods

2.1. Synthesis of Nb_2O_5 nanobelts

In a typical synthesis, 1 mmol $Nb(HC_2O_4)_5$ is added into 30 mL isopropanol, and then the mixture is stirred for 2 h and transferred into a Teflon-lined autoclave (50 mL). The autoclave is maintained at 180 °C for 48 h. Subsequently, the powder is washed and dried in a vacuum oven at 80 °C. Finally, the precursors are placed in a muffle furnace and calcined

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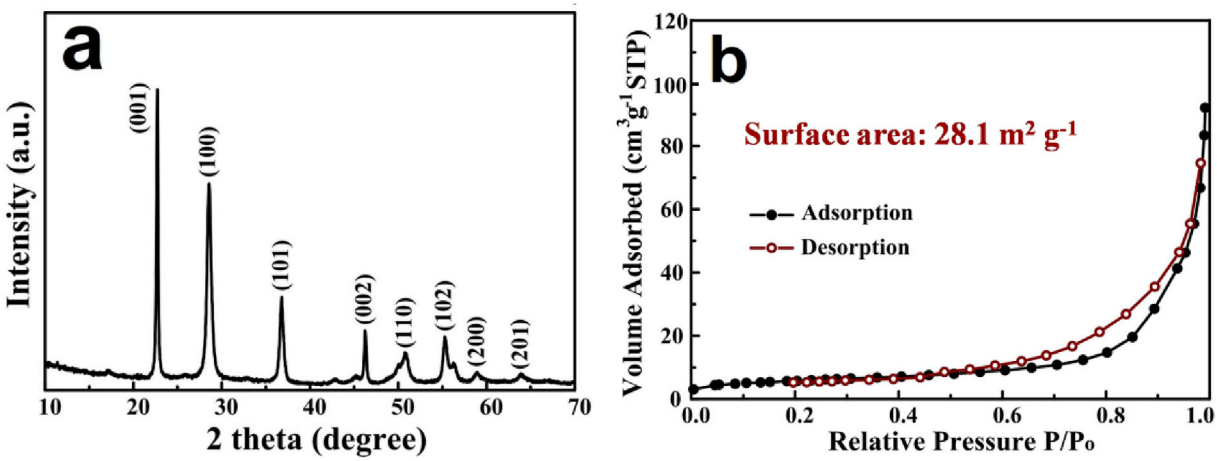

Fig. 1. (a) XRD pattern and (b) Nitrogen adsorption-desorption isotherm of the Nb_2O_5 nanobelts.

at 600 °C for 2 h to generate Nb_2O_5.

2.2. Characterizations

The phase identification of the sample is carried out by X-ray diffractometer (XRD, Rigaku D/max-2500, Cu Kα). The morphology and nanostructure of the sample are performed with field-emission scanning electron microscopy (FESEM, SU8010), transmission electron microscopy (TEM, JEM-2100F), and high-resolution TEM (HRTEM, JEM-2100F). The Brunauer-Emmett-Teller (BET) specific surface area of the sample is tested by measuring the N_2 adsorption-desorption isotherm on a Quantachrome Autosorb-IQ gas adsorption analyzer.

2.3. Electrochemical measurements

The Nb_2O_5 electrode is fabricated as follow. Nb_2O_5 nanobelts, acetylene black, and polyvinylidene fluoride (70:20:10 wt%) are dispersed into N-methyl-2-pyrrolidinone. Then, the mixture is pasted onto the Cu foils and the electrode is assembled into coin cell. Lithium is the counter and reference electrode. The electrolyte is 1 mol L^{-1} $LiPF_6$ dissolved into diethyl carbonate-ethylene carbonate (1:1 vol%). Cyclic voltammetry tests are conducted in an Electrochemical Workstation (CHI660D) at a potential window of 1.0–3.0 V. Galvanostatic tests are performed on a LAND-CT2001 system in the voltage range of 1.0–3.0 V (*vs.* Li^+/Li).

3. Results and discussion

3.1. Characterization of Nb_2O_5 nanobelts

The crystal structure of the as-synthesized Nb_2O_5 nanobelts is researched by XRD (Fig. 1a). The positions of all peaks are in good agreement with the reported data of Nb_2O_5 with a pseudohexagonal structure (JCPDS No. 07-0061, space group: *P6/mmm*). The strong peaks indicate the high crystalline nature of the sample; moreover, in comparison with the standard XRD pattern of Nb_2O_5, the (001) peak appears as the strongest one instead of the (100) peak, suggesting that the obtained Nb_2O_5 is oriented-growth [20]. Fig. 1b depicts the N_2 adsorption and desorption isotherm of the sample, and it is found that as-formed Nb_2O_5 nanobelts have a BET surface area of *ca.* 28.1 m^2 g^{-1}.

The morphologies and nanostructures of the Nb_2O_5 nanobelts are observed by FESEM, TEM, and HRTEM. As shown in the FESEM image (Fig. 2a), the sample is mainly composed of belt-like nanostructures, which are 100–200 nm in width and 0.5–1.0 μm in length; moreover, the Nb_2O_5 nanobelts have rough surfaces and they are stacked by edge-by-edge "oriented attachment" of nanorods. Similar to the FESEM result, the TEM image (Fig. 2b) indicates that the Nb_2O_5 nanobelts are constructed from parallel nanorods. The primary Nb_2O_5 nanorods have an average diameter of ~15 nm, which is in good agreement with the thickness of the Nb_2O_5 nanobelts (arrowed in Fig. 2a) and accordingly further proves the above "oriented attachment" mechanism. In the

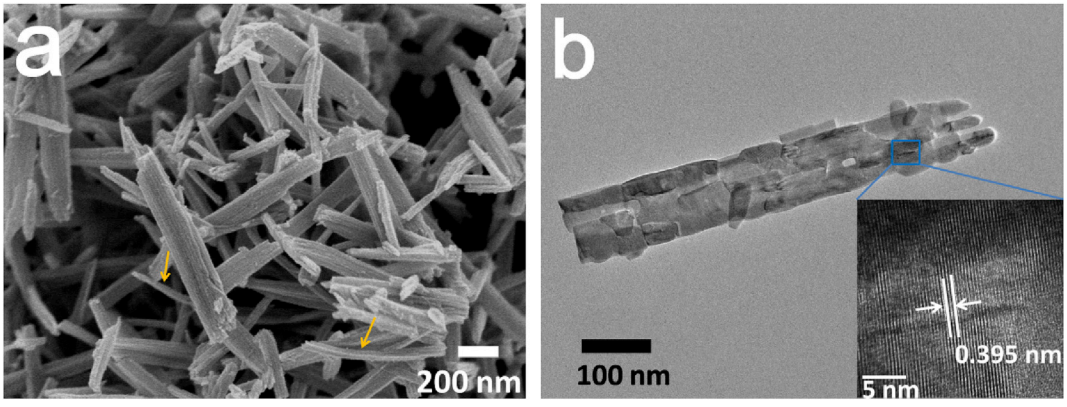

Fig. 2. (a) FESEM image and (b) TEM image of Nb_2O_5 the nanobelts, and the inset of b is the HRTEM lattice image of a typical Nb_2O_5 nanobelt originating from the blue square of b. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)