



Short communication

Improvement of the high rate capability of hierarchical structured $\text{Li}_4\text{Ti}_5\text{O}_{12}$ induced by the pseudocapacitive effect

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ABSTRACT

Hierarchical structured $\text{Li}_4\text{Ti}_5\text{O}_{12}$, assembling from randomly oriented nanosheets with a thickness of about 10–16 nm, is fabricated by a facile hydrothermal route and following calcination. It is demonstrated that the as-prepared sample has good cycle stability and excellent high rate performance. In particular, the discharge capacity of 128 mAh g^{-1} can be obtained at the high current density of 2000 mA g^{-1} , which is about 87% of that at the low current density of 200 mA g^{-1} upon cycling, indicating that the as-prepared sample can endure great changes of various discharge current densities to retain a good stability. In addition, the pseudocapacitive effect based on the hierarchical structure, also contributes to the high rate capability of $\text{Li}_4\text{Ti}_5\text{O}_{12}$, which can be confirmed in cyclic voltammograms.

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1. Introduction

Safety is one of the primary concerns in the applications of lithium-ion batteries, especially for automobile applications [1,2]. Among those safe alternatives for graphite anode, $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is of particular interest due to its extreme flat operation potential plateau at about 1.5 V vs. Li^+/Li to avoid the deposition of metallic lithium and negligible structural change during the discharge/charge process to ensure superior cycle stability [3]. However, the inherent insulating character of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ seriously limits its high rate capability, a key parameter to obtain the high power density of batteries [3–10]. To improve the high rate capability of $\text{Li}_4\text{Ti}_5\text{O}_{12}$, many attempts have been made in the past, such as introducing conductive additives and fabricating nanostructured $\text{Li}_4\text{Ti}_5\text{O}_{12}$ [3–10]. Here nanostructured $\text{Li}_4\text{Ti}_5\text{O}_{12}$ can serve to shorten the diffuse distance of lithium ion and electron, and increase contact area between electrode and electrolyte, thus improving its high rate capability [3,5,10,11]. Moreover, reducing the grain size into nanoscale can offer some further exciting features, for example, the pseudocapacitive effect existing in nanostructured TiO_2 and titanate [12–16]. The faradaic pseudocapacitive characteristic is mainly related to surface charge-transfer process, which can provide extra charge storage and faster charge/discharge rate [15,16].

In this work, we have prepared micro-sized $\text{Li}_4\text{Ti}_5\text{O}_{12}$ particles consisting with nanosheets via a facile hydrothermal route, which is an attractive architecture for the Li-ion battery electrode. The nanosheets are possible to present a pseudocapacitive effect as the interaction taking place on the surface, thus leading to improved high rate capability [14]. The particle size in the micrometer range can increase the volume energy densities compared to low-dimensional nanomaterials [11]. Furthermore, the hierarchical structured $\text{Li}_4\text{Ti}_5\text{O}_{12}$ can share the same advantages as nanostructured electrode materials as mentioned above. Thus, the as-prepared sample can be expected to present an excellent electrochemical performance as confirmed by the electrochemical tests of the galvanostatic method and cyclic voltammetry.

2. Experimental

2.1. Preparation and characterization

Precursor solution as titanium source is prepared by dissolving 6.26 g $\text{TiOSO}_4 \cdot 2\text{H}_2\text{O}$ (Chemical) in 80 ml H_2O . The as-prepared TiOSO_4 solution (5 ml) is mixed with 15 ml H_2O , and then adding dropwise into 20 ml solution that contains 0.336 g $\text{LiOH} \cdot \text{H}_2\text{O}$ (analytical) and 0.1 g urea (analytical). After stirring for 3 min, the resultant solution is transferred into a Teflon-lined autoclave (55 ml) and treated at 180°C for 24 h. The obtained precipitates were washed with original solution and about 12 ml ethanol for one time, respectively. The way of washing is key to obtain pure $\text{Li}_4\text{Ti}_5\text{O}_{12}$. After dried at 80°C for 24 h, the sample is calcined at

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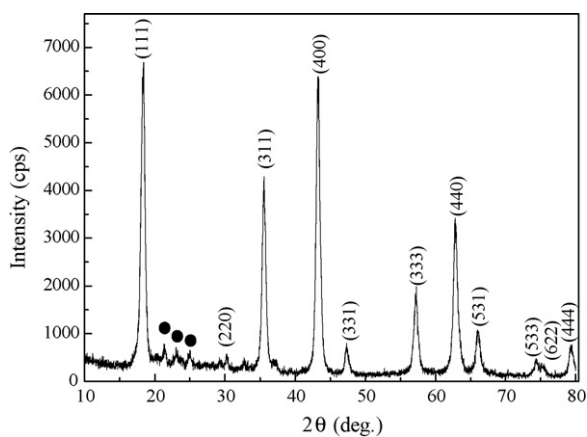


Fig. 1. XRD pattern of as-prepared $\text{Li}_4\text{Ti}_5\text{O}_{12}$. A trace of TiO_2 phase can be detected as indicated by solid circle.

500 °C for 2 h with a rate of 2°C min^{-1} in muffle furnace under Ar atmosphere. The as-prepared sample was characterized by X-ray diffraction (XRD, RIGAKU D/max-2500), scanning electron microscopy (SEM, HITACHI S-4800), and transmission electron microscopy (TEM, FEI Tecnai 20).

2.2. Electrochemical measurements

The working electrode was prepared by compressing a mixture of active materials, acetylene black, and binder (poly(tetrafluoroethylene), PTFE) in a weight ratio of 80:15:5. Lithium metal was used as the counter and reference electrodes. The electrolyte was LiPF_6 (1 M) dissolved in a mixture of ethylene carbonate (EC), ethyl methyl carbonate (EMC) and dimethyl carbonate (DMC) with a volume ratio of 1:1:1. The galvanostatic method at the charge/discharge current densities of 200, 500, 1000, 1500 and 2000 mA g^{-1} was employed to measure the electrochemical capacity and cycle life of working electrodes at room temperature using a LAND-CT2001A instrument. The cut-off potentials for charge and discharge were set at 2.5 and 1.0 V (vs. Li/Li^+), respectively. The cyclic voltammetry experiment was conducted using a CHI 600A potentiostat at a scan rate of 0.1, 0.5, 1, 2 and 4 mV s^{-1} , respectively.

3. Results and discussion

After calcination at 500 °C, crystalline spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (JCPDS 49-207) with a trace of TiO_2 phase can be detected as shown in the XRD pattern (Fig. 1). The crystallite size of $\text{Li}_4\text{Ti}_5\text{O}_{12}$, calculated by Scherrer's formula, is about 12.5 nm. The representative

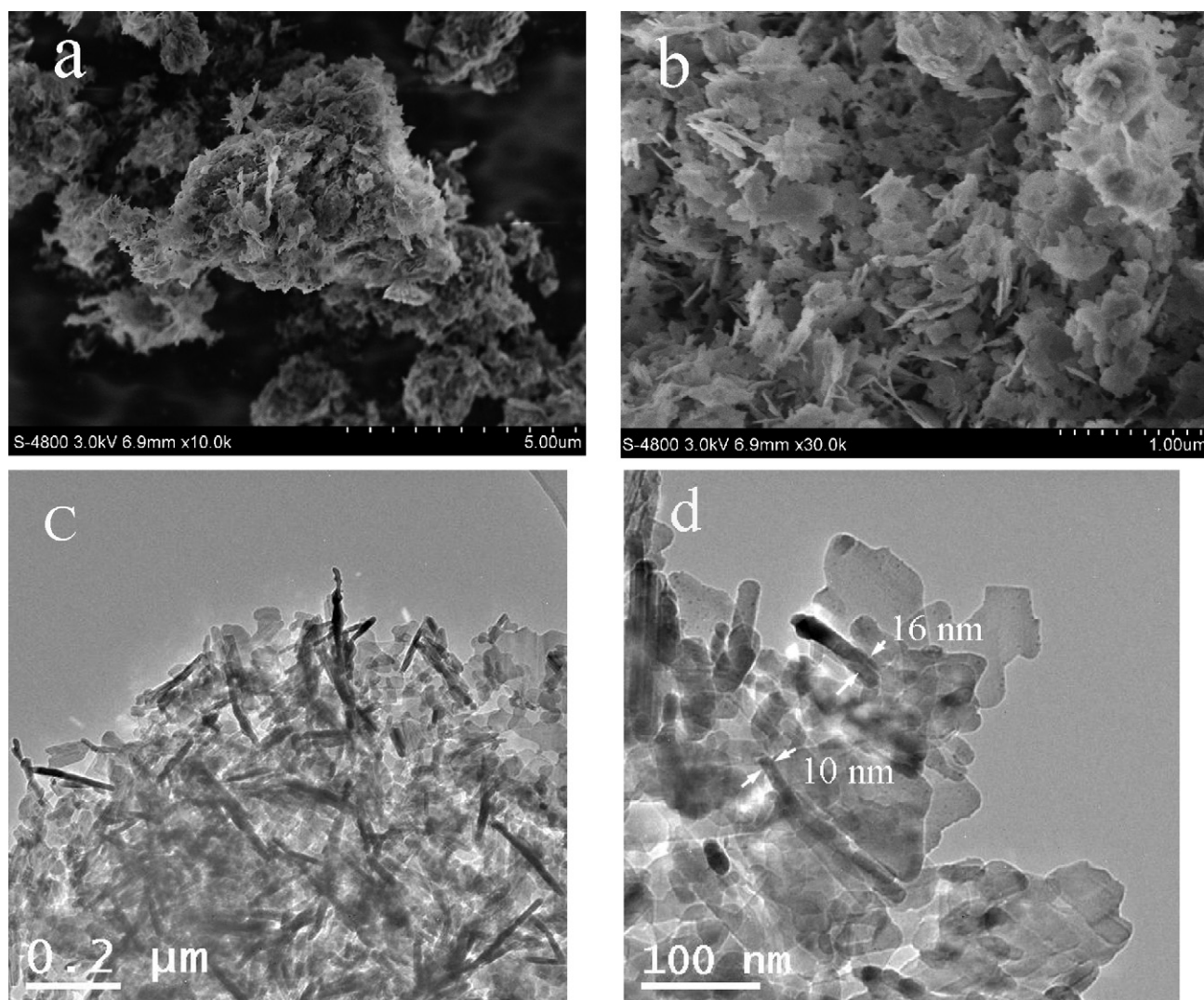


Fig. 2. SEM (a and b) and TEM (c and d) images of the as-prepared $\text{Li}_4\text{Ti}_5\text{O}_{12}$.

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