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High rate performance Fe doped lithium zinc titanate anode material synthesized by one-pot co-precipitation for lithium ion battery

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Fe doped lithium zinc titanate anode material has been synthesized via one-pot co-precipitation method for the first time. It is found that Fe element can enhance the electrochemical property of lithium zinc titanate obviously. $Li_2ZnFe_{0.05}Ti_{2.95}O_8$ particle shows largest specific capacity and best cyclic performance at high current densities among other particles. The introduction of Fe element improved electrochemical performance of the $Li_2ZnTi_3O_8$ should be owing to the enhancement of the electric conductivity and lithium ion diffusivity of $Li_2ZnTi_3O_8$.

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1. Introduction

The utilization of conventional energy source such as oil and coal has caused extensive environment problem, thus the development of clean energy has become an essential issue for human beings [1]. Graphite is usually used as anode material in commercial for lithium ion battery due to its cheap and high theory capacity (372 mAh g^{-1}). However, Graphite always suffers from safety concern because of its low charge/discharge voltage platform which results in Li dendrite. In order to solve this safety problem, Hong [2] has proposed cubic spinel structure $Li_2ZnTi_3O_8$ with a space group $P4_332$ as an anode material for LIBs in 2010 for the first time. $Li_2ZnTi_3O_8$ possesses many advantages, such as excellent cycle performance, zero strain and safety [3]. However, the inherent drawbacks of Ti-based anode material also restricted its development. For example, Low electron conductivity and lithium-ion diffusion coefficient lead to severe polarization and will hinder the achievement of high power density, thus greatly limiting the practical application of Ti-based anodes [4]. Many efforts [5–7] have been made to enhance the electronic conductivity and ion conductivity of Ti-based materials.

Fe is very abundant in earth and usually as an alien element to dope into electrode materials to improve their electrochemical performance. Yi [8] and Zhong [9] used Fe element to improve the electrochemical performance of $LiNi_{0.5}Mn_{1.5}O_4$ cathode material. The effect of Fe doping into $LiNi_{0.5}Mn_{1.5}O_4$ cathode material is mainly embodied in improvement of rate performance and enhancement of cyclic performance. In this article, we used one-pot co-precipitation method to synthesize $Li_2ZnFe_xTi_{3-x}O_8$ (x = 0, 0.03, 0.05 and 0.1) anode material for the first time.

2. Experimental

2.1. Synthesis of $Li_2ZnFe_xTi_{3-x}O_8$ (x = 0, 0.03, 0.05 and 0.1) anode material

$Li_2ZnFe_xTi_{3-x}O_8$ (x = 0.03, 0.05 and 0.1) anode material was synthesized via a modified one-pot co-precipitation method [10]. Stoichiometric of tetrabutyl titanate (TBT) was thoroughly mixed in 100 mL ethanol. Stoichiometric of $LiCH_3COO{\cdot}2H_2O$ (5% excess), $Zn(CH_3COO)_2{\cdot}2H_2O$ and $Fe(NO_3)_3{\cdot}9H_2O$ were dissolved in 50 mL deionized water, by being slowly dropped into the yellow solution under vigorous stirred. The magnetic stir sustained for 24 h, and then the precursor was transferred to 100 °C air oven to remove the solvent. The $Li_2ZnFe_xTi_{3-x}O_8$ (x = 0.03, 0.05 and 0.1) products can be obtained after 800 °C calcination for 6 h in air. $Li_2ZnTi_3O_8$ anode material can be obtained by the same method without the addition of $Fe(NO_3)_3{\cdot}9H_2O$.

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2.2. Structural characterization and electrochemical measurements

X-ray diffraction (Rigaku RINT2000) with Cu Ka radiation was used to characterize the crystal structures of the samples and recorded between 10° and 80° with a scanning speed of $3° \min^{-1}$. Scanning Electron microscope (SEM) (HitachiS4800) was used to observe the morphologies of as-prepared materials. The production of working electrode and the tests of CR2032 coin-type cell are similar to our previous research [11].

3. Results and discussions

The XRD patterns of $Li_2ZnFe_xTi_{3-x}O_8$ are shown in Fig. 1. All the samples are well crystallized in cubic structure of spinel $Li_2ZnTi_3O_8$ with $P4_332$ space group. It is notable that no other impurity peaks have been appeared in the XRD patterns and all of them are in good agreement with the standard $Li_2ZnTi_3O_8$ (JCPDF# 86-1512). After Fe doped, the peak position variation of (1 1 1) plane of all samples are magnified and shown in Fig. 1(b). With the increase of the amount of Fe, the peaks of (1 1 1) plane gradually shift to higher 2Θ angle (according to the equation: $\lambda = 2d\sin\Theta$). The structural analyses were conducted on the recorded XRD data based on Rietveld refinement. As shown in Fig. 1(c–f), all the samples are cubic, and the value of a for $Li_2ZnFe_xTi_{3-x}O_8$ ($x = 0$, 0.03, 0.05 and 0.1) are 8.3687 Å, 8.3685 Å, 8.3643 Å and 8.3616 Å, respectively. This result can be attributed to the ion radium of Fe^{3+} (0.055 nm) is smaller than Ti^{4+} (0.061 nm) which also illustrated that Fe element has doped to Ti site. ICP analysis has been used to further identify

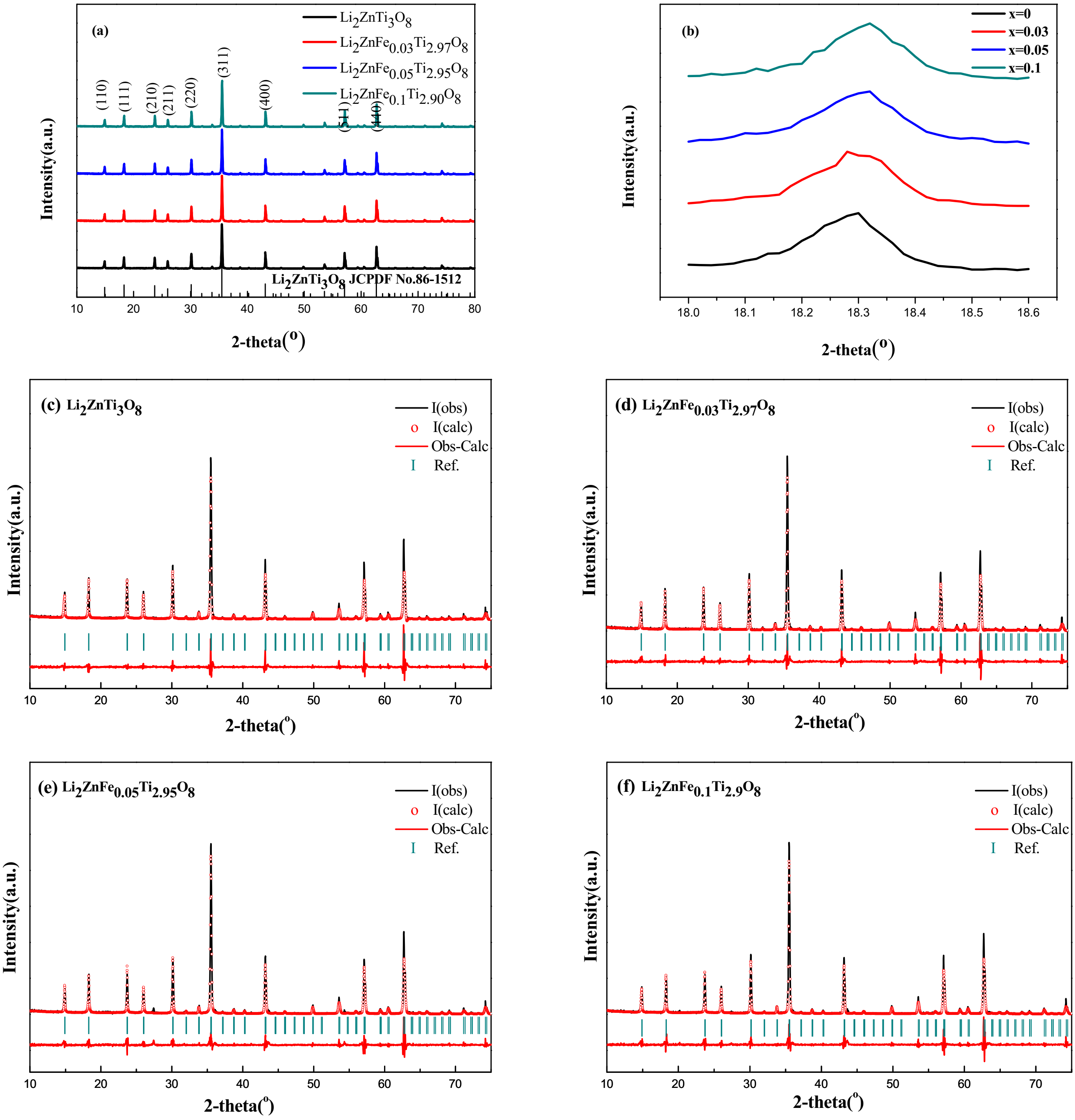

Fig. 1. XRD patterns (a) enlarged (1 1 1) peaks (b) and Rietveld refinement of $Li_2ZnFe_xTi_{3-x}O_8$ ($x = 0$, 0.03, 0.05 and 0.1) (e–f).

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