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Preparation and electrochemical performance of tantalum-doped lithium titanate as anode material for lithium-ion battery

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Abstract: The electrochemical performance of Ta-doped $Li_4Ti_5O_{12}$ in the form of $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ was characterized. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to characterize the structure and morphology of $Li_4Ti_{4.95}Ta_{0.05}O_{12}$. Ta-doping does not change the phase composition and particle morphology, while improves remarkably its cycling stability at high charge/discharge rate. $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ exhibits an excellent rate capability with a reversible capacity of 116.1 mA·h/g at 10C and even 91.0 mA·h/g at 30C. The substitution of Ta for Ti site can enhance the electronic conductivity of $Li_4Ti_5O_{12}$ via the generation of mixing Ti^{4+}/Ti^{3+} , which indicates that $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ is a promising candidate material for anodes in lithium-ion battery application.

Key words: lithium-ion battery; lithium titanate; anode material; doping

1 Introduction

The ever-growing demand for portable batteries with high energy density is exerting pressure for the development of advanced lithium-ion battery. For large-scale applications such as electric and hybrid vehicle systems, the vital issue is the availability of advanced materials. In recent years, there has been considerable effort devoted to developing high energy density, safe and reliable new materials to use as the anodes in lithium-ion battery [1]. The spinel Li₄Ti₅O₁₂ has been found to be an attractive anode material for lithium-ion battery [2-4]. In the lithium titanate spinel-type structure of Li₄Ti₅O₁₂, the formal valence of titanium is +4, which is the highest achievable oxidation state possible for titanium. This Li₄Ti₅O₁₂ material has been found to intercalate lithium ions without strain or shrinkage to the lattice. It has a flat Li insertion potential at about 1.55 V (versus Li^+/Li), above the reduction potential of common electrolyte solvents, mitigating the formation of solid-electrolyte interface (SEI) and avoiding formation of lithium dendrites to make the battery safer. However, several disadvantages exist compared with graphite, including the poor electric conductivity that limits its full capacity at high charge-discharge rates.

Effort has been expended to improve the conductivity. Several methods were proposed: 1) doping $Li_4Ti_5O_{12}$ with other metal cations or non-metal ions in Li, Ti or O sites [5–11]; 2) incorporating a second phase with high electronic conductivity such as carbon and metal powder [12–14]; 3) making a nitridation to form oxynitride species on its surface [15]. However, to our best knowledge, there are less investigations on the electrochemical characteristics of Ta-doped $Li_4Ti_5O_{12}$ as an anode material. In this work, we proposed to partially substitute Ti^{4+} with Ta^{5+} , which will cause a transition of a certain amount of Ti^{4+} to Ti^{3+} as charge compensation. The transition from Ti^{4+} to Ti^{3+} in $Li_4Ti_5O_{12}$ will lead to an increase in the electronic conductivity and thus improve the rate performance.

2 Experimental

2.1 Preparation of materials

 $Li_4Ti_5O_{12}$ samples were prepared using a solid-state method from TiO₂ (anatase structure) and LiOH·H₂O. The Ta-doped $Li_4Ti_5O_{12}$ was prepared also using a solid-state method with TiO₂ (anatase structure), LiOH·H₂O and Ta₂O₅. In both cases, mass fraction excess LiOH·H₂O was used to compensate for lithium volatilization during the high temperature heating. A doping level of 1% (mole fraction) Ta was selected,

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which yields the following composition, Li₄Ti_{4.95}Ta_{0.05}O₁₂, assuming that Ta sits on a Ti site. This is probably a good assumption in that the ionic radius of Ta^{5+} is 0.064 nm, similar to the ionic radius of Ti^{4+} (0.061 nm) and much smaller than the ionic radius of Li^+ (0.076 nm). Powders of the precursor materials were mixed in a mortar and pestle with enough methanol to form slurry. The undoped mixed reactant mixture was heated at 800 °C for 5 h in air (oxidizing) and the Ta-doped mixed reactant mixture was heated at 800 °C for 5 h in 3% (volume fraction) H₂/Ar (reducing) to obtain the final powders.

2.2 Characterization

The crystal structure of the powders was characterized by X-ray diffraction (XRD, Rigaka Dmax-RB) using Cu-K_{α} radiation (10° $\leq 2\theta \leq 90^{\circ}$). Cell parameters were refined with the program FULLPROF [16]. Scanning electron microscope (SEM, KYKY2800) was used to study the morphology of the materials.

2.3 Electrochemical tests

The electrochemical cycling performances of the Li₄Ti₅O₁₂ powders were evaluated at room temperature (20 °C) with laboratory-scale Li/Li₄Ti₅O₁₂ button cells including a lithium metal foil as counter electrode, a composite of 80% (mass fraction) Li₄Ti₅O₁₂, 10% (mass fraction) acetylene black (AB), and 10% (mass fraction) polytetrofluornethelene (PTFE) binder as a cathode. A micro-porous polypropylene film (Celgard 2400) was used as a separator and 1 mol/L LiPF6 solution with the 1:1 in volumetric ratio of ethylene carbonate to dimethyl carbonate (EC-DMC) was used as the electrolyte. All cells were assembled inside a glove box filled with ultra-pure argon. Charge-discharge characteristics of the cells were recorded in the potential range of 1.0-3.0 V using a LAND batteries test system (CT2001A, Jinnuo Electronics Co., Ltd., Wuhan, China) and specific capacities were calculated based on the mass of Li₄Ti₅O₁₂. Electrochemical impedance spectrum (EIS) was also measured using a potentiostat/galvanostat EG&G 273A coupled to a frequency response analyzer (FRA) EG&G 1025. The impedance data were obtained between 1 MHz and 10 mHz at 5 mV as the applied sinusoidal perturbation. Electrical resistance measurement of the sintered and polished pellets of Li₄Ti₅O₁₂ was also performed at electrochemical workstation above.

3 Results and discussion

3.1 Phase characteristics

The color of the synthesized Li₄Ti₅O₁₂ powder is

white. The XRD patterns of the Li4Ti5O12 and Li₄Ti_{4.95}Ta_{0.05}O₁₂ are shown in Fig. 1(a). All the sharp diffraction peaks can be attributed to the cubic spinel structure of Li₄Ti₅O₁₂ without obvious impurity phase, which indicates that Ta5+ has successfully entered the lattice of the spinel and do not change its structural characteristics. The XRD pattern reveals that the (111) peak of the Li₄Ti_{4.95}Ta_{0.05}O₁₂ shifted to smaller angels. For a clear observation, the peak position variation of (111) plane is magnified and shown in Fig. 1(b). The XRD refinement according to the Rietveld method indicates that the lattice parameter of Li₄Ti_{4.95}Ta_{0.05}O₁₂ is 8.368 Å and that of the $Li_4Ti_5O_{12}$ is 8.362 Å [17], which are consistent with results reported by WOLFENSTINE and ALLEN [10]. The doping of Ta will cause the lattice constant to become large. This may be caused by the reason that the substitution of Ta5+ for Ti4+ site will cause the transition of a certain amount of Ti⁴⁺ to Ti³⁺ as charge compensation [10], which will cause the increase of the lattice constant of the $Li_4Ti_5O_{12}$ because $Ti^{3+}(0.067 \text{ nm})$ is larger than Ti⁴⁺ (0.061 nm). For heat-treatment under reducing conditions, according to charge neutrality



Fig. 1 XRD patterns of $Li_4Ti_5O_{12}$ and the $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ (a) and magnified (111) peaks of $Li_4Ti_5O_{12}$ and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ (b)

condition (Kroger-Vink notation [16]) is given in Eq. (1) and hence, an increase in electronic conductivity occurs [10].

$$[e']=[Ta_{Ti}\cdot] \tag{1}$$

where an electron, e', corresponds to Ti^{3+} on a Ti^{4+} site.

3.2 Morphology

Figure 2 shows the SEM images of the undoped $Li_4Ti_5O_{12}$ powders and the Ta-doped powders. The grains of pure $Li_4Ti_5O_{12}$ are small with the size generally distributed in the range of 200–300 nm. Small particles will enlarge the contact areas between grains and electrolyte, and thus improve the specific capacity of the electrode. In Fig. 2(b), the grain size of the Ta-doped powders is larger than the pure one, but is also nano-sized.



Fig. 2 SEM images of $Li_4Ti_5O_{12}$ (a) and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ (b)

The acquisition of these homogeneously nano-sized $Li_4Ti_5O_{12}$ powders could be attributed to the precursor material, nano-sized anatase structure TiO_2 , which could be mixed more sufficiently with the other materials compared with the usually used micro-sized rutile structure TiO_2 . The sintering time in this work is much shorter than the ordinarily reported 10–15 h. Short sintering time was also critical to prevent the grains from excessive growth.

3.3 Electrochemical performance

The electrochemical properties of the powders are determined by charge-discharge test at constant current

density. Figure 3 shows the first charge-discharge curves of the undoped and Ta-doped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ powders at 0.1*C* rate in potential window between 3.0 and 1.0 V. The cycling behavior is typical of LTO with a flat plateau at an average potential of 1.55 V which is attributed to a two-phase phenomenon pertaining to $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{Li}_7\text{Ti}_5\text{O}_{12}$ phases. The voltage platform is the result of an adjustment of the above intercalation and deintercalation processes. The sharp and linear increases of the voltage at the end of the charge and the start of the discharge curves are results of electrode polarization. The cation distribution in $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{Li}_7\text{Ti}_5\text{O}_{12}$ phases during electrochemical charge-discharge processes could be written as follows [2]:

$$[Li]_{8a} [Li_{1/3}Ti_{5/3}]_{16d} [O_4]_{32e} + Li^+ + e = [Li_2]_{16c} [Li_{1/3}Ti_{5/3}]_{16d} [O_4]_{32e}$$
(2)



Fig. 3 Initial discharge-charge curves of $Li_4Ti_5O_{12}$ and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ at 0.1*C* rate

During the first discharge, undoped Li₄Ti₅O₁₂ takes about 2.78 Li atoms, corresponding to a capacity of 161.9 mA·h/g that matchs well with the expected theoretical capacity for Li4Ti5O12. However, the Ta-doped Li₄Ti₅O₁₂ takes about 2.69 Li atoms, corresponding to a capacity of 156.7 mA·h/g, less than the undoped one. A small electrode polarization of about 0.02 V is observed between charge-discharge curves, indicating the existence of good interparticle electrical contacts and ion transport. In Li₄Ti₅O₁₂ structure, 75% Li^+ ions locate at tetrahedral 8a sites, 25% Li^+ and Ti^{4+} are randomly distributed at octahedral 16d sites, and O^2 ions occupy the 32e sites, the 8b, 48f and 16c sites are empty [2]. During lithiation process, 3 Li-ions can be accommodated by Li4Ti5O12, which will make spinel Li₄Ti₅O₁₂ transform to rock-salt Li₇Ti₅O₁₂. In this process, all Li-ions, including those in 8a site and the newly inserted ones, will move and occupy the 16c site, and

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then all octahedral sites are filled. Ta-doping may impede the transportation of Li-ions from 8a to 16c sites, influencing the insertion of outside Li-ions into $\text{Li}_4\text{Ti}_5\text{O}_{12}$ structure. As a consequence, the capacity of Ta-doped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is decreased.

High rate performance is one of the most important electrochemical characteristics of lithium ion batteries for HEV application. The charge-discharge capacities of pure Li₄Ti₅O₁₂ and Ta-doped Li₄Ti₅O₁₂ electrodes at different current rates from 0.1C to 30C are shown in Fig. 4. It can be seen that, the rate performance of Li₄Ti₅O₁₂ can be improved by doping Ta. The charge and discharge capacities decrease with increasing the current rate. Li₄Ti_{4.95}Ta_{0.05}O₁₂ exhibits an excellent rate capability compared with Li4Ti5O12, although Li4Ti5O12 shows much more initial capacity than Li4Ti4.95Ta0.05O12. At 0.1C, Li₄Ti_{4.95}Ta_{0.05}O₁₂ presents a discharge capacity of 152.8 mA·h/g, while the $Li_4Ti_5O_{12}$ exhibits a discharge capacity of 161.9 mA·h/g. At 30C, the discharge capacity of Li₄Ti_{4.95}Ta_{0.05}O₁₂ still remains 91.0 mA·h/g, while the capacity of the undoped Li₄Ti₅O₁₂ is only 44.5 mA·h/g. We proposed to partially substitute Ti⁴⁺ with Ta⁵⁺, which will cause a transition of a certain amount of Ti⁴⁺ to Ti³⁺



Fig. 4 Initial discharge-charge curves of $Li_4Ti_5O_{12}$ (a) and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ (b) at different current rates (0.1*C*, 1*C*, 5*C*, 10*C*, 20*C*, 30*C*)

as charge compensation. The transition from Ti^{4+} to Ti^{3+} in $Li_4Ti_5O_{12}$ will lead to an increase in the electronic conductivity and thus improve the rate performance.

The cycling performance of the Ta-doped and undoped $Li_4Ti_5O_{12}$ samples at the rate of 1*C* is exhibited in Fig. 5. In Fig. 5, more than 11.1% of the capacity loss occurs to the undoped $Li_4Ti_5O_{12}$ at 1*C* after 100 cycles, while the Ta-doped $Li_4Ti_5O_{12}$ loses only 6.3% after 100 cycles. The 100th discharge capacity of the Ta-doped $Li_4Ti_5O_{12}$ electrode is 146.8 mA·h/g, but the corresponding value of the $Li_4Ti_5O_{12}$ electrode is decreased to 144.7 mA·h/g. This indicates that the introduction of Ta ions can increase the cycle stability. This suggests that the electrode polarization is probably decreased by Ta-doping.



Fig. 5 Cycling performance of $Li_4Ti_5O_{12}$ and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ samples (current rate of 1*C*)

Electrochemical impedance spectroscopy (EIS) may be considered one of the most sensitive tools for studying the changes in the electrode behavior due to surface modification. EIS results of the coin cells with the $Li_4Ti_5O_{12}$ and Ta-doped $Li_4Ti_5O_{12}$ cathodes are shown in Fig. 6. The measurement was carried out after the cells have been discharged to the depth of 50% followed by three cycles. The impedance spectra are composed of one semicircle at higher frequencies followed by linear part at lower frequency end. The semicircle in the high region represents the migration of the lithium ions at the electrode/electrolyte interface. The low frequency region of the straight line is attributed to the diffusion of the lithium ions into the bulk of the electrode material, the so-called Warburg diffusion [18].

The relationship between the imaginary impedance and the low frequencies is governed by Eq. (3). The diffusion coefficients of the lithium ions in the bulk electrode materials were calculated by Eq. (4). The Warburg impedance coefficient can be obtained from the straight lines [17–18]. The relation is governed by Eq. (5). These calculated results are recorded in Tabe1. The real impedance of the $Li_4Ti_5O_{12}$ and the $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ samples in the low frequencies is illustrated in Fig. 7.



Fig. 6 EIS results of $Li_4Ti_5O_{12}$ and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ samples

$$Z'' = -\sigma_{w} \omega^{-1/2} \tag{3}$$

$$D=0.5\left(\frac{RT}{c\sigma_{\rm w}AF^2}\right)^2\tag{4}$$

$$Z_{\rm re} = R_{\rm s} + R_{\rm ct} + \sigma_{\rm w} \omega^{-1/2} \tag{5}$$

where Z" is the imaginary impedance; ω is the angular frequency in the low frequency region; R_{ct} is the charge-transfer resistance; R_s is the electrolyte resistance; D is the diffusion coefficient; R is the gas constant; T is the thermodynamic temperature; F is the Faraday's constant; A is the area of the electrode surface; c is the molar concentration of Li⁺; δ_w is the Warburg impedance coefficient [18–19].

Table 1 Impedance parameters of $Li_4Ti_5O_{12}$ and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ samples

Sample	$R_{\rm s}/\Omega$	$R_{\rm ct}/\Omega$	$\sigma_{\rm w}/(\Omega \cdot {\rm cm}^2 \cdot {\rm s}^{-1/2})$	$D/(\mathrm{cm}^2\cdot\mathrm{s}^{-1})$
Li ₄ Ti ₅ O ₁₂	15.67	55.58	22.65	1.78×10^{-9}
$Li_4Ti_{4.95}Ta_{0.05}O_{12}$	8.234	45.23	11.24	1.12×10^{-8}



Fig. 7 Real impedance of $Li_4Ti_5O_{12}$ and $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ samples in low frequencies

The obtained diffusion coefficients show that Ta-doped $Li_4Ti_5O_{12}$ sample has higher mobility for Li^+ diffusion than the undoped $Li_4Ti_5O_{12}$ sample. This is attributed to the concentration of electrons caused by the transition of a certain amount of Ti^{4+} to Ti^{3+} as charge compensation. The transition from Ti^{4+} to Ti^{3+} in $Li_4Ti_5O_{12}$ will lead to an increase in the electronic conductivity and thus improve the rate performance.

4 Conclusions

1) $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ powders have been successfully synthesized by a simple solid state reaction. XRD patterns show that the $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ has a good crystallinity and a high phase purity.

2) The $Li_4Ti_{4.95}Ta_{0.05}O_{12}$ electrode presents better cycling performance than the $Li_4Ti_5O_{12}$ electrode prepared by the similar process. Ta-doping does not change the phase composition and particle morphology, while improves remarkably its cycling stability at high charge-discharge rate.

3) The substitution of Ta for Ti site can enhance the electronic conductivity of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ via the generation of mixing $\text{Ti}^{4+}/\text{Ti}^{3+}$. At 30*C*, the discharge capacity of the $\text{Li}_4\text{Ti}_{4.95}\text{Ta}_{0.05}\text{O}_{12}$ still remains at 91.0 mA·h/g, while the capacity of the undoped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is only 44.5 mA·h/g. More than 11.1% of the capacity loss occurs to the undoped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ at 1*C* after 100 cycles, while the Ta-doped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ loses only 6.3% after 100 cycles. The 100th discharge capacity of the Ta-doped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ electrode is 146.8 mA·h/g, but the corresponding value of the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ electrode is decreased to 144.7 mA·h/g.

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锂离子电池极材料钽掺杂钛酸锂的 制备及电化学性能

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摘 要:通过固相合成制备了钽掺杂材料 Li₄Ti_{4.95}Ta_{0.05}O₁₂。通过 XRD 和 SEM 来表征 Li₄Ti_{4.95}Ta_{0.05}O₁₂的结构和 形貌。 钽掺杂并没有改变本体材料的结构和形貌,而且显著提高了材料的循环性能和倍率性能。Li₄Ti_{4.95}Ta_{0.05}O₁₂ 在 10C 和 30C 倍率时的放电容量分别是 116.1 mA·h/g 和 91.0 mA·h/g。Ta 掺杂取代了 Li₄Ti₅O₁₂ 中的 Ti 的位置, 产生了 Ti⁴⁺/Ti³⁺混合价态,从而提高了钛酸锂的电导率。故具有优异的高倍率性能,是一种优异的锂离子电池负 极材料。.

关键词: 锂离子电池; 钛酸锂; 负极材料; 掺杂

(Edited by YANG Hua)