

Electrochemical Behavior of Gallium-Doped Lithium Titanate

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Abstract

A synthesis method of gallium-doped lithium titanate is proposed. The effect of gallium-doping on electrochemical properties of the $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ is studied in detail. It has been established that gallium doping results in the increased cycling stability of lithium titanate. The good electrochemical performance of the gallium-doped lithium titanate can be attributed to the increase conductivity and effect diffusion coefficient.

Keywords: Gallium doping, lithium titanate, lithium-ion battery, energy density.

1. Introduction

Nowadays lithium titanate is regarded as a promising anode material for lithium-ion batteries. It is conditioned by its ability to operate at increased current densities up to 20 C. Theoretical capacity at the insertion of three lithium ions per one formula unit of lithium titanate amounts to about 175 mAh g⁻¹, in this case the charge-discharge curve represents a plateau in the area of potentials of 1.55 V, which is conditioned by the existence of two phases: delithiated ($\text{Li}_4\text{Ti}_5\text{O}_{12}$) and lithiated ($\text{Li}_7\text{Ti}_5\text{O}_{12}$) [1,2].



Small change in the size of a cubic cell of this compound (from 8.3595 to 8.3538 Å [3]) determines high cycling stability of the material. The number of charge-discharge cycles can go up to several thousand without significant degradation [4].

Relatively low electronic and lithium conductivity (10^{-13} S cm⁻¹) [5-8], as well as low discharge capacity, which is worse than that of graphite and substantially worse than that of silicon or tin [2], can be qualified as disadvantages of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. However, these disadvantages are largely compensated by high cycling stability, including at high charge/discharge currents.

In order to increase electronic and lithium conductivity, there were attempts of heterovalent doping of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ with divalent (Cu^{2+}), trivalent (Cr^{3+} , Sc^{3+} , Al^{3+} , Tb^{3+}) and quintavalent (Ta^{5+}) cations [9-15]. In a number

of cases, increased conductivity of obtained materials was observed but electrochemical properties of the doped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ were not studied.

2. Experimental

2.1. Synthesis of gallium-doped lithium titanate

Gallium-doped lithium titanate was synthesized using the citrate method. Titanium tetrabutylate (99%, Alfa Aesar) and lithium carbonate (99%, Fluka) were dissolved in the ethanol-nitric acid mixture (volume ratio 5:1), and solutions of gallium (99.99%, Aldrich) in nitric acid and citric acid (98%, Sigma) were added in the minimum quantity of water. Lithium carbonate was taken with a 5% surplus to prevent possible losses of lithium during subsequent annealing at high temperatures. The obtained mixture was heated sequentially at 95°C during 24 hours and at 250°C during 5 hours. The so formed precursor was ground in an agate mortar to a smooth paste that was subjected to final annealing at 800°C during 5 hours in air.

2.2. Physical characterization and electrochemical measurements

The crystal structure was characterized by X-ray diffraction (XRD) with the $\text{CuK}\alpha$ radiation performed on Rigaku D/MAX 2200 diffractometer. The Rigaku Application Data Processing software package was used for spectra processing. The microstructure of obtained samples was analyzed with Carl Zeiss NVision 40 scanning electron microscope.

2.2 Electrode preparation and cell assembling

The study of electrochemical properties of lithium titanate samples was conducted in three-electrode sealed electrochemical cells with a lithium auxiliary electrode and a lithium reference electrode. Working electrodes were

prepared using a standard paste method. Stainless steel mesh was used as a current collector. The active mass was prepared by mixing the lithium titanate powder (80% wt), carbon black (15% wt, Timcal, Belgium) and polyvinylidenefluoride (5% wt, Aldrich) predissolved in N-methylpyrrolidone (Aldrich). The mass of active material in the electrode was about $8\text{--}10\text{ mg cm}^{-2}$. Electrodes were pressed under a pressure of $1,000\text{ kg cm}^{-2}$ with subsequent vacuum drying at 120°C . Electrochemical cells were assembled in an argon-filled glove box (“Spectro-systems”, Russia). The 1 M LiPF_6 in a mixture of ethylene carbonate-diethyl carbonate-dimethyl carbonate (1:1:1) (all components of the electrolyte were qualified as ‘extra dry’ and were purchased from Aldrich) was used as an electrolyte. Water content in the electrolyte did not exceed 10 ppm. Polypropylene nonwoven fabric of $20\text{ }\mu\text{m}$ (NPO “Ufim”) was used as a separator.

3. Results and discussion

3.1. Characterization of gallium-doped lithium titanate

X-ray patterns of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ samples contain reflexes of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ only (Card No. 72-0426 PDF2 database), which evidences that the obtained material is single-phase. Initially, the formula of $\text{Li}_{4+z}\text{Ti}_{5-z}\text{Ga}_z\text{O}_{12}$, where z is the degree of doping, was proposed for gallium-doped lithium titanate. Additional weak lines inherent in Li_2TiO_3 can be detected in $\text{Li}_{4+z}\text{Ti}_{5-z}\text{Ga}_z\text{O}_{12}$ at $z = 0.2$ [16]. An additional point is that all X-ray patterns display a lines shift to the high-angle region, which evidences of a decreased lattice parameter. In fact, with the growth of the degree of doping, the a parameter decreases from $8.3596(6)$ for $\text{Li}_4\text{Ti}_5\text{O}_{12}$ to $8.3537(4)$ for $\text{Li}_{4+z}\text{Ti}_{5-z}\text{Ga}_z\text{O}_{12}$.

It can be conditioned by the fact that radii of cations of Ga^{3+} , Ti^{4+} and Li^+ are similar in size, that is why gallium ions can get inserted into both, positions of titanium, and positions of lithium. Ga^{3+} ion attracts oxygen ions more intensively than Li^+ ion considering Coulomb interaction, which results in a grater lattice contraction. To verify this assumption, the structure of the doped sample was updated using the Rietveld method. Since the sample of doped lithium titanate with the degree of doping 0.2 contains small quantity of impurities (Li_2TiO_3), and the degree of doping is rather low, the structure was updated for the material of $\text{Li}_{4+z}\text{Ti}_{5-z}\text{Ga}_z\text{O}_{12}$ composition at $z=0.1$. The same tendency in the distribution of gallium was supposed to be preserved for the degree of gallium doping $z=0.2$. According to the data obtained as a result of updating the structure using the Rietveld method of the sample of $\text{Li}_{4+z}\text{Ti}_{5-z}\text{Ga}_z\text{O}_{12}$ composition at $z=0.1$, gallium ions

occupy both, positions of lithium (8a), and positions of titanium (16d). Moreover, there are about 2.5 times less of gallium ions in octahedral sites than in tetrahedral ones. In accordance with these results, the formula of gallium-doped lithium titanate should be as follows: $\text{Li}_{4+x-3y}\text{Ti}_{5-x}\text{Ga}_{x+y}\text{O}_{12}$ ($x+y=0.1\text{--}0.2$; $y/x=2.56$). The obtained data agree with the observed dependence of lattice parameters on the doping level. Thus, when the doping degree are $x+y=0.1$ or $x+y=0.2$, the formulas are as follows: $\text{Li}_{3.812}\text{Ti}_{4.972}\text{Ga}_{0.1}\text{O}_{12}$ and $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$.

According to the data from the scanning electron microscopy, synthesized samples of $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ represent a rather homogeneous crystalline mass (Fig. 1). Growth steps are clearly seen in the micrographs. The particle surface becomes smoother due to blurred facets. The size of particles for the undoped compound varies within the range of $400\text{--}500\text{ nm}$, for $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ – $600\text{--}770\text{ nm}$ (Fig. 1). Following the results of electron probe microanalysis, there are cations of titanium, gallium and oxygen in all the studied areas of the sample. For doped titanate, the ratio of Ga/Ti is $0.2/5.2$, which is almost the same as the initial load.

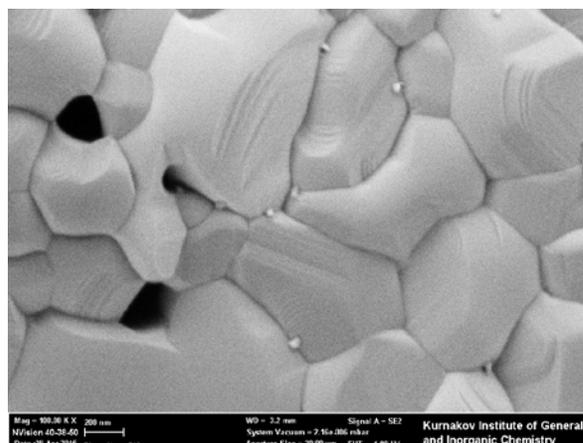


Fig. 1. Scanning electron microscopy of the sample of $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$.

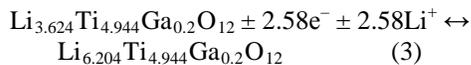
3.2 Electrochemical insertion and extraction of lithium in $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ and $\text{Li}_4\text{Ti}_5\text{O}_{12}$

During the study of lithium titanate in a three-electrode electrochemical cell with a lithium metal counter electrode, the cathode process of lithium insertion corresponds to charging of lithium titanate, and the anode process of lithium extraction – to discharging of lithium titanate in lithium-ion batteries. At the first stage, lithium insertion and extraction processes in the standard range of potentials ($3 - 1\text{ V}$) were studied.

Charge-discharge curves of plain and doped lithium titanate contain well-defined sites in the area of potentials

of 1.55 V (Fig. 2a), which reflect the lithium insertion and extraction process by a two-phase mechanism. The irreversible capacity at the first cycle for all studied samples was 13–16%, but at the second cycle the coulombic efficiency of the cycling for all samples went up to 97%. The discharge capacity of undoped lithium titanate at the current density of 20 mA g⁻¹ was about 155 mAh g⁻¹. For the doped sample the discharge capacity was less – about 147 mAh g⁻¹. Lowering of the discharge capacity of the doped sample is explained by the fact that unlike titanium gallium cannot participate in a redox-process. The theoretical specific capacity of Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ is about 171 mAh g⁻¹ (against 175 mAh g⁻¹ for plain lithium titanate). At higher current densities the discharge capacity of the doped sample exceeds the capacity of Li₄Ti₅O₁₂ (Fig. 2b). Thus, at the current density of 3,200 mA h g⁻¹ the discharge capacity of Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ doubles the discharge capacity of Li₄Ti₅O₁₂. It should be also noted that after the high-current density cycling and upon return to low current, the discharge capacity of all samples almost coincides with the value registered at the first cycles. It evidences of low cycling degradation of the samples. The calculated values of degradation for all samples did not exceed 0.03 % per cycle.

According to the equation (1), the reversible insertion of one lithium ion corresponds to the specific capacity of about 58 mAh g⁻¹. Thus, it is fair to say that brutto-reactions take place at the current density of 20 mA g⁻¹ in the range of potentials from 1 to 3 V:



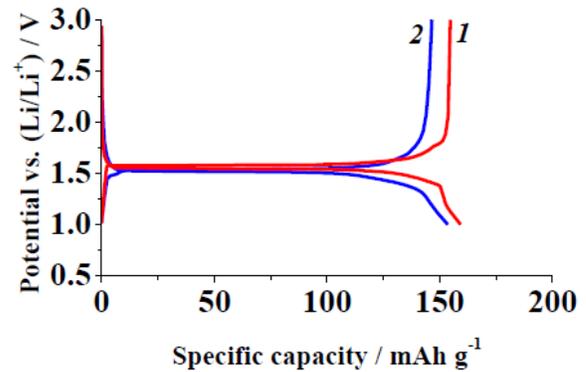
According to electrochemical data, after the charge at the current density of 20 mA h g⁻¹ in the range of potentials from 3 to 1 V undoped lithium titanate has an average composition of Li_{6.64}Ti₅O₁₂. It implies that approximately 12% of the material remain uncharged. For Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ this portion is 14%. Besides, even at a full-charge phase of Li₇Ti₅O₁₂ two more lithium ions can be reduced to oxidation state 3⁺. Thus, a reversible process of lithium insertion-extraction can be continued at potentials more negative than 1V.

Additional information about reversibility of the lithium insertion-extraction process can be obtained from cyclic voltamograms.

Cyclic voltamograms of Li₄Ti₅O₁₂ and Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ at different potential scan rates in the interval of potentials from 1 to 3 V are shown in Fig. 3. The curves of both materials have clear peaks that reflect the lithium insertion and extraction process, while the

forms of the curves of plain and doped lithium titanate differ.

a



b

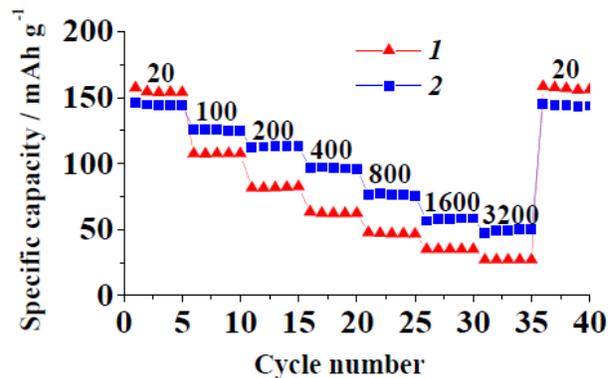
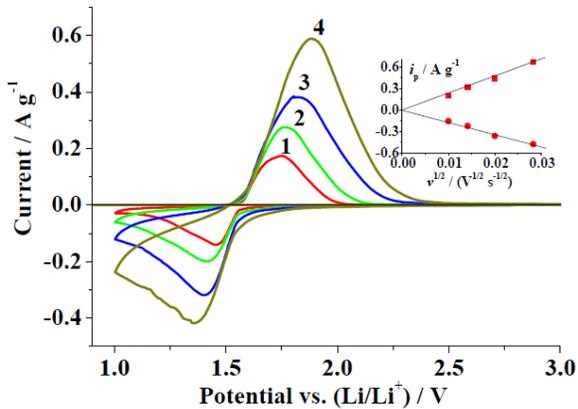


Fig. 2. (a) Charge-discharge curves at the current density of 20 mA g⁻¹ and (b) dependence of the discharge capacity on the current density (see values in the Fig.) at the cycling in the range of potentials of 3.0-1.0 V for Li₄Ti₅O₁₂ (1) and Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ (2)

Cyclic voltamograms of Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ have narrower peaks and larger values of specific currents in the peaks of voltamograms. The difference between the potentials of anode and cathode peaks (ΔE) of electrode materials is determined by the reversibility of the lithium insertion-extraction process and specific resistance of phases' materials that are formed during charge and discharge processes. For Li_{3.624}Ti_{4.944}Ga_{0.2}O₁₂ ΔE at different potential scan rates, ΔE ranged from 85 to 247 mV, while for Li₄Ti₅O₁₂ it ranged from 293 to 523 mV. The data from cyclic voltamograms evidence of a greater reversibility of the lithium insertion-extraction process for

doped lithium titanate. It should be concluded that increased reversibility is the consequence of increased conductivity of the doped sample against the undoped one.

a



b

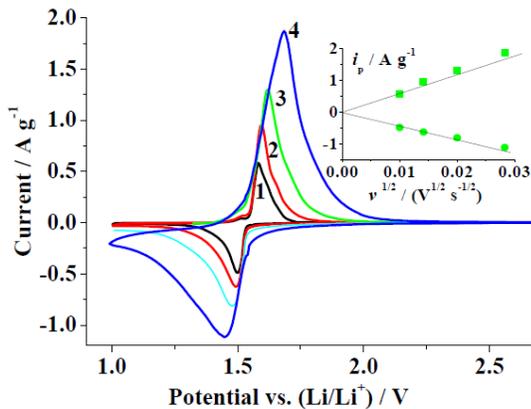


Fig. 3. Cyclic voltamograms of the samples of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (a) and $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ (b) in the range of potentials from 1 to 3 V. Potential scan rates ($\mu\text{V s}^{-1}$): 1 – 100, 2 – 200, 3 – 400, 4 – 800

The dependences of the current in the peaks of voltamograms on the square root of the potential scan rate, shown in the insert in Fig. 3, are linear and go through the origin of coordinates, which evidences of diffusion control of the process of lithium insertion-extraction in plain and doped lithium titanate.

The calculated data (Table 1) obtained from cyclic voltamograms are in good agreement with the data from galvanostatic measurements, namely, at a small potential scan rate (0.1 mV s^{-1}) the discharge capacity of plain lithium titanate slightly exceeds the discharge capacity of doped lithium titanate. Simultaneously, the situation is reverse when the potential sweep scan increases. Thus, when $v=0.4 \text{ mV s}^{-1}$ the discharge capacity of $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ exceeds the discharge capacity of

$\text{Li}_4\text{Ti}_5\text{O}_{12}$, and when $v=0.8 \text{ mV s}^{-1}$ the discharge capacity of $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ is 1.5 times higher than the discharge capacity of $\text{Li}_4\text{Ti}_5\text{O}_{12}$.

Table 1. Calculated data for $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ obtained from cyclic voltamograms

	Samples	Potential scan rate, mV s^{-1}			
		0.1	0.2	0.4	0.8
Capacity in cathode half-cycle, mAh g^{-1}	$\text{Li}_4\text{Ti}_5\text{O}_{12}$	118	104	99	79
	$\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$	142	130	117	108
Capacity in anode half-cycle, mAh g^{-1}	$\text{Li}_4\text{Ti}_5\text{O}_{12}$	122	110	98	78
	$\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$	125	122	116	107
ΔE , mV	$\text{Li}_4\text{Ti}_5\text{O}_{12}$	293	357	428	523
	$\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$	85	100	147	247

One of the explanations of higher values of the discharge capacity may be an increased lithium effective diffusion coefficient (D_{eff}) at gallium doping of lithium titanate. To confirm this assumption, D_{eff} for both materials were calculated based on the data from cyclic voltamograms during the cycling at different ranges of potentials.

The Randles–Sevcik equation was used for calculation:

$$D = i_p / v^{1/2} * 2.69 * 10^{-5} * S * c \quad (4)$$

where v is the potential scan rate (V/s), S is the true area of the surface (cm^2), and c is the concentration of lithium ions.

At the cycling in a narrow range of potentials (1.0 – 3.0 B), the concentration of lithium ions for $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ amounted to 0.022 and 0.0196 mole cm^{-3} , accordingly. The effective diffusion coefficient at the insertion of lithium ions into $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ was $7.35 * 10^{-10}$ and $1.65 * 10^{-9} \text{ cm}^2 \text{ s}^{-1}$, accordingly. At the extraction of lithium the effective diffusion coefficient of lithium ions from $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ amounted to $1.48 * 10^{-9}$ and $7.0 * 10^{-9} \text{ cm}^2 \text{ s}^{-1}$, accordingly.

4. Conclusions

Gallium-doped lithium titanate was synthesized using the citrate method. According to the data obtained as a result of updating the structure using the Rietveld method of the sample of $\text{Li}_{4+z}\text{Ti}_{5-z}\text{Ga}_z\text{O}_{12}$ composition at $z=0.1$, gallium

ions occupy both, positions of lithium (8a), and positions of titanium (16d). Moreover, there are about 2.5 times less of gallium ions in octahedral sites than in tetrahedral ones. In accordance with these results, the formula of gallium-doped lithium titanate should be as follows: $\text{Li}_{4+x-3y}\text{Ti}_{5-x}\text{Ga}_{x+y}\text{O}_{12}$ ($x+y=0.1-0.2$; $y/x=2.56$). Thus, when the doping degree is $x+y=0.2$, the formula is as follows: $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$. Electrochemical measurements have proved ability of gallium-doped lithium titanate to operate without degradation. In this case the discharge capacity of $\text{Li}_{3.624}\text{Ti}_{4.944}\text{Ga}_{0.2}\text{O}_{12}$ was about 148 and 50 mAh g^{-1} at current density of 20 and 3200 mA g^{-1} , respectively. In the same time undoped lithium titanate cannot operate at current densities higher than 800 mA g^{-1} .

Acknowledgments

The research was financially supported by the Russian Foundation for Basic Research (Project No. 14-29-04068 ofi_m).

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