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## Li<sup>+</sup> - INTERCALATION IN TALC DOPED BY OXYGEN AND SULFUR

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The influence of talc doping by oxygen and sulphur on the characteristics of Li<sup>+</sup>-intercalation current formation is investigated. This doping cause changes of crystal and electronic structure of material, which is in accordance with the results of X-ray structural analysis, impedance spectroscopy and to the discharge curves form. It defines different ways of intercalation process parameters change. Modification of talc by oxygen allows improving of discharge characteristics of the lithium power sources that have cathodes based on the obtained materials.

**Keywords:** Li<sup>+</sup>-intercalation, talc, cathode material.

### Introduction

During the last decade [1-5], much more attention attracted by searching of effective cathode materials for electrochemical sources with lithium anode. Mentioned materials should correspond to many demands such as presence of "host" position system with certain energy of lithium cation intercalation, good defusing characteristics, chemical and electrochemical stability, topochemistics. Nevertheless, at the same time properties of "host"-materials electronic structure are ignored. Such disregard of electronic structure can be explained for metallic and semi-metallic phases. Of course, model of determined zones works sufficiently for these phases [6]. As power sources specific capacitance (with intercalation mechanism of current formation reactions) determined by amount of "host" positions, so from the viewpoint of mentioned model its size will depend on the width of the conductance band and the degree of its occupation. But as it is shown [7] such structures do not provide enough high capacitance therefore desire to increase it results in use non-metallic phases as cathodes [4, 8, 9].

From the other side researchers pay attention not only to the well-known cathode materials for power sources but also to other ecologically safe and cheap materials. The main requirement to these materials is high electric capacitance of mass and volume unit. Natural minerals with layered and channel structure and with dielectric or semiconductor conductive mechanism realization are partially meet these requirements. The important possibility of realization in layered talc lithium-cation current formation reaction has been shown in the work [10] and phase-thermodynamic analysis of the formed intercalation composition was given. Efforts to improve specific capacitance characteristics of these current sources by halogen co-intercalation, which was used for graphite, bismuth selenide [11] or titanium disulphide [12], did not give expected results. Quite the contrary it becomes clear that it is impossible to gain success without understanding influence of electronic structure on the intercalation current formation. Unfortunately, there were not enough purposeful and systematic researches for this branch. Some researches [13-15] just proved topicality of it. That is why the aim of this article is an attempt to fill the knowledge base gap, using the example of natural layered mineral – talc.

### 1. Experimental part

In the experiments has been used talc from Chinese fields (Figure 1 contains photography of its microstructure). Existence of certain admixtures (MgO, Mg(NO<sub>3</sub>)<sub>2</sub> [16]) and structural defects, energy states of which are located not far away from the conductance zone bottom, facilitate effective realization of intercalation-cation current formation reactions.

With a purpose of certain change of energetic topology system of own defects origin high dispersed talc (middle size of particles ~ 5 μm) was heated in vacuum quartz vessels to the temperature of 150°C and

500°C with the rate of heating 2°C/m and 5°C/m respectively with the next ageing at these temperatures during 90 minutes. After that vessels were filled with oxygen and annealed at mentioned temperatures 15, 90 and 180 m. For sulphur intercalation (pure fraction for analysis), it was put into the vessel, and heating process was performed at the same temperature values. Room temperature has been reached in the regime of turned out oven.

X-ray structural researches of compressed samples were made on the diffractometer DRON-3 in  $\text{CuK}\alpha$  - radiation ( $\lambda=1.5418 \text{ \AA}$ ).

For electrochemical researches electrodes from active material (talc), current conductive addition (acetylen soot), bonding agent in mass relation 85% : 10% : 5% on nickel substract (square  $2 \text{ cm}^2$ ) were formed. Talc mass was not more than 30 mg. Electrochemical cell was formed from the researched electrode, lithium counter electrode and comparative electrode that were dipped in 1 m solution  $\text{LiBF}_4$  in  $\gamma$ -butyrolactone. Thermodynamic and kinetic regularities of lithium intercalation of origin and modified talc were researched by impedance spectroscopy method in the frequencies range  $10^{-3} \div 10^6 \text{ Hz}$  with the help of measuring complex AUTOLAB-100 (ECO CHEMIE) completed with computer software FRA-2.



Fig. 1 Microphotography of talc samples

Comparing of high-dispersed talc compressed under the pressure 100 Bar diffraction patterns to etalon diffraction patterns of ICSD catalogue showed that researched talc belongs to the structure modification with the spatial group C-1 (triclinic system) and applied pressure did not cause significant structure changes (Figure 2). This structural modification of talc is characterized by the following parameters:  $a=5,298 \text{ \AA}$ ,  $b=9,173 \text{ \AA}$ ,  $c=9,352 \text{ \AA}$ ,  $\alpha=90,46$ ,  $\beta=98,68$ ,  $\gamma=90,09$ . Period of talc identity in the interlayer direction (axis C) is  $9,352 \text{ \AA}$ . Half width of diffraction line for the plane 003 was  $0,05 \text{ \AA}$ .

## II. Results and discussion

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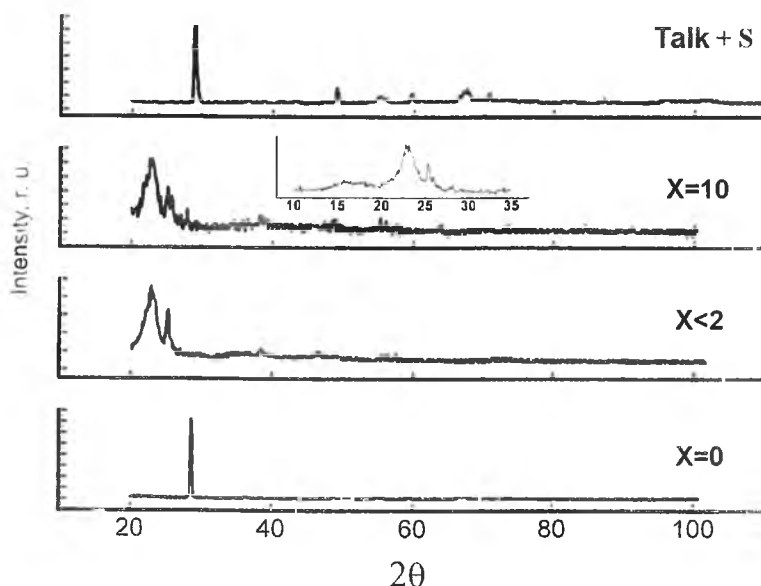


Fig. 2. X-ray diffraction patterns of talc: not intercalated ( $x=0$ ); the degree of intercalation not less than 2 ( $x < 2$ ); the degree of intercalation 10 ( $x=10$ ); not intercalated by doped sulphur (Talc+S)

Lithium intercalation even by low amount (molar lithium loading  $x < 2$ ) cause significant changes within talc structure (Figure 2). Interplanar distance 003 increases from  $3,117 \text{ \AA}$  to  $3,530 \text{ \AA}$ . The period of

Identification in the interlayer direction increases from 9,352 Å to 10,590 Å accordingly. Also half width of diffraction line (003) increases from 0,05 Å to 0,11 Å. At high concentration of intercalated lithium ( $x \sim 10$ , Figure 2) diffraction line fission (003) of intercalated talc (addition to the Figure 2,  $x \sim 10$ ) was observed. That could be caused by lithium ions localization in the different structural position of crystal matrix.

Annealing of origin talc in the oxygen atmosphere at the temperature 150°C did not made significant structure changes in contrast to the annealing at 500°C. Diffuse halo with the maximum at  $d=4.61$  Å appears in the low angle zone of diffraction pattern. This appearance indicates partly amorphization of talc that could be connected with destruction of crystal structure and single fragments reduction. Changes in the interlayer distance were not encountered during the measurement with accuracy to 0,005 Å.

Annealing at the same conditions at the atmosphere of sulphur results in the transformation of crystal lattice – interlayered distance reduced to 0,02 Å (Figure. 2) that often happens during the intercalation process [17, 18].

Discharge curves of the elements with cathode based on researched materials are given (Figure 3).

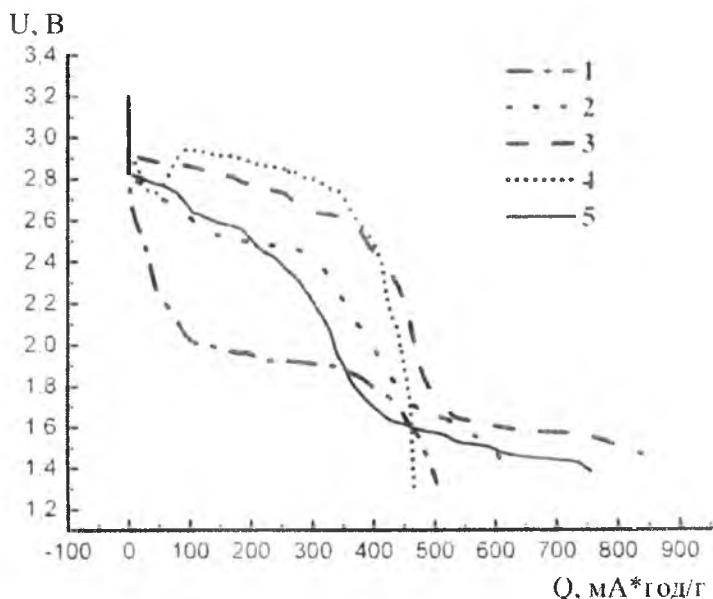


Fig. 3. Discharge curves of lithium elements with cathodes on the base of talc annealed in the oxygen atmosphere 1.5 hour (curve 1), oxygen 0.25 hour (curve 2), 1.5 hour (curve 3), 3 hour (curve 4) and origin talc (curve 5)

Comparing them with the data of X-ray structural analysis tells that the interlayer distance change values are not accordable to the Gibbs energy change values of the lithium intercalation process  $\Delta G(x)$ . Independence of thermodynamic parameters from the degree of distortion of crystal lattice is clear, and can be shown easily with comparing of origin talc X-ray diffraction patterns to talc doped by sulphur and oxygen. Small changes of diffraction patterns after annealing in the sulphur steam results in huge  $\Delta G(x)$  increasing, but significant disordering of atomic planes after annealing in the oxygen atmosphere will modify thermodynamic parameters not considerably. Therefore we can suggest that annealing of talc in different atmospheres and modes will change mainly its own defective energetic spectrum. So, the modification of the electronic states energetic topology near the Fermi level is the reason of Gibbs energy maximum of lithium intercalation. Under the conditions of independence of intercalated atoms and matrix layers interaction energy ( $E_0$ ) formation connection from the degree of intercalation  $x$  ( $E_0(x)=const$ ) change of chemical potential of intercalated atom  $\mu_i(x)$  can be given as a sum of entropy contribution filling “host” positions, energy interaction between host components, changes of regions near Fermi level and power contribution which is needed for moving apart matrix layers, accordingly to the equation [19]:

$$\mu_i(x) = kT \ln|x/(1-x)| + Z\omega x + [E_f(x) - E_f(0)] + L\partial C/\partial x + E_0 \quad (1)$$

In this equation  $k$  – Boltzman constant,  $Z$  – number of the closest host positions;  $\omega$  – interaction energy of intercalated host components;  $E_f$  – position of Fermi level,  $C$  – distance between layers,  $L$  – coefficient that determined by Lennard-Johnson potential function. Take into consideration known [6] relation for electromotive force of Faradaic current formation

$$\Delta \bar{G} - eU(x) = \mu_l(x) - \mu_0 \quad (2)$$

where  $\Delta \bar{G}$  - molar Gibbs energy of reaction,  $U(x)$  - electromotive force of reaction  $\text{Li}^+$  intercalation,  $e$  - electron charge,  $\mu_l(x)$  - chemical potential of intercalated lithium in talc,  $\mu_0$  - chemical potential of lithium at the metal anode. It is easy to notice that structure of discharge curves (excluding initial stage of intercalation for which main contribution is given by first and fourth equation members) will form by concentration dependence of interaction energy in the subsystem of intercalant and Fermi level position changes. Last factor will depend on energetic topology of own defects system through the mediation of  $E_f(0)$  and its indirect influence on  $E_f(x)$ . Electron affinities of oxygen and sulphur are 1,467 eV and 2,077 eV [20] respectively. That fact determines different changes of Fermi level  $E_f(0)$  position during intercalation of them into the talc. This situation will cause different character of  $\mu_l(x)$  changes and discharge voltage. In the mentioned host loading concentrated ranges strong difference in the structure of discharge curves is observed (Figure 3).

We need to pay attention on the following two aspects to investigate the admixture states influence on the process thermodynamic parameters:

1. Task to increase intercalation capacity in the given voltage range is similar by its nature to the task of fixing level Fermi which is solved by doping of certain admixtures in radiation physics [21]. In our case the certain admixtures can not only create the additional states under Fermi level, but also they define behavior of  $E_f(x)$  and difference  $\mu_l(x) - \mu_0$ .

2. Main advantage of intercalation current formation is topochemistry of intercalation reactions. That is why the practical importance of discharge curve horizontal type should be determined by phase transition not due to chemical interaction of host component with a matrix, but as a result of attractant interaction with the intercalated lithium. It is obvious that the last mentioned interaction type also will facilitate the increase of host loading degree. To find out conditions of appearance this interaction model Hamiltonian system is described as:

$$\bar{H} = H_0 + \bar{H}_{\text{int}} + \bar{H}_{h-g} \quad (3)$$

where:

$$H_0 = \sum_{\alpha, \kappa, \sigma} E_{\alpha, \kappa, \sigma} C_{\alpha, \kappa, \sigma}^+ C_{\alpha, \kappa, \sigma} \quad (4)$$

host material Hamiltonian ( $\alpha$  enumerates valence band and conductive zone);

$$\begin{aligned} H_{\text{int}} = & E_I \sum_{\sigma} (a_{\sigma}^+ a_{\sigma} + b_{\sigma}^+ b_{\sigma}) + U(n_{a\uparrow} n_{a\downarrow} + n_{b\uparrow} n_{b\downarrow}) - \frac{1}{2} \bar{A} (n_{a\uparrow} n_{b\uparrow} + n_{a\downarrow} n_{b\downarrow}) \\ & + I \sum_{\sigma} (a_{\sigma}^+ b_{\sigma} + h e) + W \sum_{\sigma, \zeta} (n_{a\sigma} n_{b\zeta}) - W \sum_{\sigma} (n_{ab} + n_{bc}) + W_{\text{ion}} \end{aligned} \quad (5)$$

host subsystem Hamiltonian, selected in the way of equivalent Hamiltonian Heitler-London [22];

$$H_{h-g} = \sum_{\alpha, \kappa, \sigma} (V_{\alpha, \kappa} a_{\sigma}^+ C_{\alpha, \kappa, \sigma} + h e) + \sum_{\alpha, \kappa, \sigma} (\bar{V}_{\alpha, \kappa} b_{\sigma}^+ C_{\alpha, \kappa, \sigma} + h e) \quad (6)$$

where  $V_{\alpha, \kappa} = V_{\alpha, \kappa} \exp(i\vec{k}\vec{d}_z)$ ,  $V_{\sigma, \kappa} = - \int \varphi_{\alpha}(\vec{r} - R_1) \frac{e^2}{|\vec{r} - \vec{R}_1|} \psi_{\alpha, \kappa}(\vec{r}) d^3\vec{r}$ ,  $\varphi_{\alpha}(\vec{r} - R_1)$  - monoatomic wave

function,  $\psi_{\alpha, \kappa}(\vec{r})$  - Bloch function that describes host component interaction with the matrix charge carrier.

Interaction between two intercalated atoms of lithium in one or nearby host positions results in breaking up their hybridized orbital on binding and non-binding branches with power position accordingly under and above Fermi level. Such topology of intercalant power levels indicates the expedience of Heitler-London transformation model in the method of molecular orbitals to simplify the single-electron states finding. Then, adding new operators:

$$\hat{A}_{\sigma} = \frac{1}{2} (a_{\sigma} + b_{\sigma}); \hat{A}_{\sigma}^+ = \frac{1}{2} (a_{\sigma}^+ + b_{\sigma}^+); \hat{B}_{\sigma} = \frac{1}{2} (a_{\sigma} - b_{\sigma}); \hat{B}_{\sigma}^+ = \frac{1}{2} (a_{\sigma}^+ - b_{\sigma}^+), \quad (7)$$

in Hartree-Fock approximation for modified Hamiltonian (3) we get:

$$\bar{E}_1 = E_I - W - |I| + M_1; \bar{E}_1 = E_I - W + |I| + M_2, \quad (8)$$

and

$$M_1 = \sum_{\alpha, k} \frac{|V_{\alpha, k}|^2 \cos^2 \frac{\vec{k}\vec{d}}{2}}{\omega - E_{\alpha, k}}; \quad M_2 = \sum_{\alpha, k} \frac{|V_{\alpha, k}|^2 \sin^2 \frac{\vec{k}\vec{d}}{2}}{\omega - E_{\alpha, k}}; \quad (9)$$

where  $E_i$  – electron intercalation atom energy,  $W = \frac{e^2}{|\vec{R}_1 - \vec{R}_2|}$  Coulomb interaction, between two

electrons,  $I$  – hybridization energy.

Condition of attraction between introduced lithium atoms and formation of related states is found:

$$\bar{E}_1 \langle n_{A\uparrow} \rangle + W + E_c + (E_c - \langle n_{A\uparrow} \rangle) < 2E_F$$

or

$$E_F > E_i - W - |I| + M_1 + T \ln \frac{W}{E_c - E_i + |I| - M_1} \quad (10)$$

From (10) it is clearly obvious, that even at nonzero temperature attracting of the intercalated lithium atoms is possible thus without phonons participation. Such attracting represents the desired nature of 1 type phase transition, and its realization can be promoted by alloying. Frequency dependences of elements impedance with cathodes on the basis of experimental materials showed that processes with diffusion-kinetic control are determinative. The Nyquist diagrams have a typical for the mentioned processes form (Figure 4) that gives reasons for the choice of processes equivalent scheme as it is presented in Figure 5.

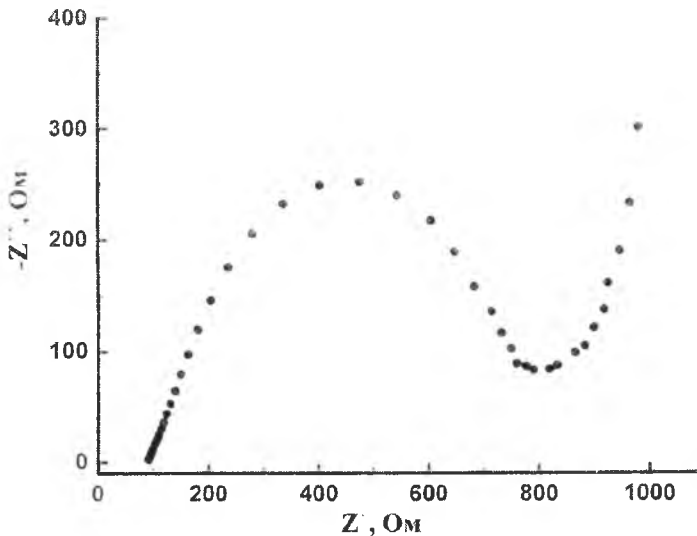


Fig. 4. The look of Nyquist diagrams of researched elements

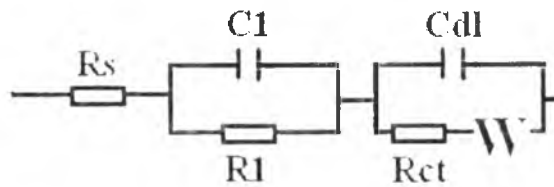


Fig. 5 Equivalent electric scheme of lithium intercalation process in cathodes on the base of researched materials

On this scheme  $R_s$  is internal resistance of the system,  $R_1||C_1$  link reflects passivation processes on a lithium anode, and  $C_{dl}||R_{ct}-W$  link – processes on a cathode, there  $R_{ct}$  is resistance of the charge transfer stage through the bound of electrolyte | talc,  $C_{dl}$  is a capacity of double electric layer on this bound,  $W$  is an element that determines lithium diffusion into a matrix. Values of diffusion coefficients are presented in the Table 1. They are calculated by the program Zwiev-2 taking into account the half of diameter of charge accumulator particle according to [23, 24].

Values of diffusion coefficient of lithium in cathodes based on researched materials (where talc I and talc II materials ageing at the oxygen atmosphere 0.25 hour and 3 hour accordingly)

Material	Diffusion coefficient $D \cdot 10^{12}$ , $\text{sm}^2/\text{s}$
Talc	1.04
Talc I	0.13
Talc II	0.12

As we can see from Table 1 modification of talc by oxygen results in decreasing of lithium diffusion coefficient which can be caused [25] by changes amount of vacant positions, the height of power barrier and energy of interaction lithium with matrix. First two factors are determined by the changes in the material structure of talc and its partial amorphisation. The last factor is determined by the change of electronic structure of material that is proved by results of X-ray structural analysis and the form of discharge curves (Figure 2, 3).

## Conclusions

According to the results of the X-ray structural analysis talc intercalation even by low amount of lithium results in an increase of interplanar (003) distance from 3,117 Å to 3,530 Å. Annealing of talc in the oxygen atmosphere at the temperature of 150°C does not cause noticeable changes in the structure; annealing at the temperature of 500°C results in its partial amorphization that probably related to the decomposition of crystalline structure and separate fragments reduction. At last, annealing in the sulphuric atmosphere with those conditions cause the interlayer distance reduction (0,02 Å)

The doping of talc by oxygen and sulphur with different time of ageing causes the change of thermodynamic and kinetic parameters of lithium intercalation process that is related to the changes of structural and electronic properties of talc according to the results of X-ray structural analysis, impedance spectroscopes and types of discharge curves. From the practical point of view an element on the basis of talc annealed in the atmosphere of oxygen during 3 hours has the best power characteristics. It resulted in increasing of specific capacitance value to 400  $\text{mA} \cdot \text{hour}/\text{g}$  and power to 1200  $\text{W} \cdot \text{hour}/\text{kg}$ . These values for the origin talc are 250  $\text{mA} \cdot \text{hour}/\text{g}$  and 650  $\text{W} \cdot \text{hour}/\text{kg}$  respectively.

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