Technology of preparation of the LiCoO$_2$ reactive mass for chemical current sources

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Abstract. The article discusses methods for the preparation of chemically reactive mass LiCoO$_2$ in the form of dispersion for use in chemical current sources. The dispersion technique and the method of deposition of LiCoO$_2$ particles from the obtained dispersion on the electrode material are described. The results of SEM on the penetration of LiCoO$_2$ into the material and the results of mass changes are presented.

1. Introduction

Most of the lithium-ion electric batteries used in portable electronics are made using a positive electrode from lithium cobalt oxide. Complex oxide LiCoO$_2$ has a layered structure in which lithium and cobalt ions are ordered in alternating planes. The presence of planes occupied exclusively by lithium ions provides the possibility of almost complete extraction of the alkali metal and, thus, the applicability of this compound as a cathode material in chemical current sources (CCS). From the point of view of increasing the capacity of the CCS, the use of lithium cobalt oxide together with electrode materials based on sorbed carbon fiber having a stable highly developed surface, low weight and high conductivity is promising [1–3].

In the present work, the possibilities of impregnation of metallized carbon fiber «Busofit-UL-50» using the dispersion of LiCoO$_2$ in propylene carbonate (PC) or n-methylpyrrolidone (NMP) are considered. Metallization Busofit held to reduce the equivalent series resistance between the elements of the CCS [4–6].

2. Grinding of LiCoO$_2$

Grinding of lithium cobalt oxide was carried out in a bead mill manufactured by Netzsch, type Labstar. A grinding chamber with a volume of 600 ml was used, which was 70% filled with yttria-stabilized zirconia (YSZ) beads and with a diameter of (0.4+0.6) mm. 600 ml of a liquid diluent (NMP or PC) were poured into the bowl of the mill, then 500 g of lithium cobalt oxide were introduced in portions of 60 g, after which 200 ml of solvent were added until the working volume of the mill was filled. The grinding was carried out at a rotation speed of the main shaft of 3000 rpm and a pumping speed of 120 rpm for 8 hours, the main particle size distribution from 100 to 300 nm was obtained from the measurement results.
To measure the size, samples were taken with a volume of 1 ml, and then dried on an electric stove at a temperature of 200 °C to completely remove the liquid diluent. The dried powder was dispersed in PC and NMP using an ultrasonic bath for 15 minutes. The measurement was performed in ethylene glycol. The measurement results are presented in figure 1.

![Figure 1](image1.png)

**Figure 1.** Particle size distribution after 5 hours, 7 hours and 8 hours of grinding.

3. **Impregnation of textile Busofit with LiCoO$_2$**

The resulting dispersions were impregnated in a metallized carbon textile to penetrate the dispersion as deep as possible into the material.

The impregnation process was carried out by spreading the dispersion on the surface of the carbon material and further lamination and recorded on a scanning electron microscope (SEM). Figure 2 shows the image of metallized Busofit, obtained by using SEM.

![Figure 2](image2.png)

**Figure 2.** Metallized Busofit.

After impregnation of Busofit with dispersion LiCoO$_2$ + PC, the PC began to be gradually absorbed into the material and after 1 minute almost completely went deep, except for some small droplets that
adhered on individual hairs. LiCoO$_2$ partially went deep, but the bulk of it froze on the surface of the material. Figure 3 shows a photograph of a filament of carbon material impregnated with a drop of dispersion of LiCoO$_2$ + PC and a graph of the composition of the material deposited on the fiber. The obtained data with SEM show that dispersion made it possible to obtain the required particle size of LiCoO$_2$ less than 300 nm and there are no excess impurities in the formed coating.

During the impregnation of LiCoO$_2$ + NMP in Busofit, the bonds between the fiber instantaneously collapse and the upper layer of the material is instantly broken. LiCoO$_2$ partially went deep into the material. Figure 4 shows a photograph with an SEM filament of a carbon material impregnated with a dispersion of LiCoO$_2$ + NMP and X-ray spectral analysis of the composition of the material. X-ray spectral analysis did not reveal any third-party impurities in the formed coating.

The metallized Busofit sample with a mass of $m_0$ and 3x3 cm in size was weighed on an analytical scale, then a drop of dispersion was squeezed out of the syringe and smeared over the material, after which the sample was weighed ($m_1$). Then a drop of dispersion was squeezed out onto the sample at the same place again and the sample was weighed again ($m_2$). The process was repeated until the 3rd drop.
According to the results of 3 spreads and weighings, a sample with LiCoO$_2$ + NMP impregnation gained 54.32% of the initial mass of metallized Busofit without impregnation, and a sample with LiCoO$_2$ + PC impregnation gained 41.81% of the initial weight. The measurement results are presented in table 1.

**Table 1.** The change in the mass of the electrode material after lubrication.

| Weight | LiCoO$_2$ + NMP | LiCoO$_2$ + PC |
|--------|-----------------|----------------|
| $m_0$  | 0.2218          | 0.2231         |
| $m_1$  | 0.2508          | 0.2441         |
| $m_2$  | 0.3291          | 0.2814         |
| $m_3$  | 0.3423          | 0.3165         |

4. Impregnation of textile Busofit with LiCoO$_2$ in vacuum

To penetrate the obtained dispersions even further into the carbon fiber, vacuum impregnation of metallized Busofit was carried out. Figure 5 shows images of deposited LiCoO$_2$ on carbon fiber filaments after vacuum impregnation.

![Figure 5.](image)

(a) – vacuum impregnation with dispersion of LiCoO$_2$ + NMP; (b) – vacuum impregnation with dispersion of LiCoO$_2$ + PC.

5. Conclusions

The obtained results allow drawing the following conclusions:

1. A dispersion with a particle size of LiCoO$_2$ less than 300 nm was obtained.
2. LiCoO$_2$ with a thickness of less than 500 nm partially settled on the fiber of metallized Busofit, when using dispersion with PC, however, the bulk of the active mass remained on the surface.
3. The dispersion of LiCoO$_2$ + NMP destroyed the weaving of filaments on the surface of the material and went deep into. LiCoO$_2$ almost completely covered the surface of the fiber.
4. Measurement of the mass of the material showed that the amount of LiCoO$_2$ deposited in Busofit after spreading the dispersion of LiCoO$_2$ + PC increased by 41.81%. After spreading LiCoO$_2$ + NMP, the mass of the material increased by 54.32%. The increase in mass due to the penetration of the active material into the material allows predicting an increase in the energy intensity of the CCS.
5. Vacuum impregnation made it possible to fill the pores of the metallized Busofit and evenly apply LoCoO$_2$ over the entire surface of the fibers.

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