

## **Phase Transition in Li-Mn Spinels; *in situ* XRD and Impedance Spectroscopy Analysis**

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Lithium-manganese stoichiometric spinel  $\text{LiMn}_2\text{O}_4$  undergoes upon cooling phase transformation. In the present work stoichiometric and  $\delta$ -spinels synthesized by sol-gel technique [1] and commercially available ones were studied at  $-25$  to  $+100^\circ\text{C}$  by *in-situ* XRD and impedance spectroscopy (in freq. Range 0.1 Hz to 10 MHz). Rietveld as well as separate peaks (400) analysis were performed for phase and structure identification. The correlation of the XRD profile parameters and conductivity was demonstrated. Additional phases in commercial objects seemingly containing only one phase were found and described. The lattice parameters of the regular and orthorhombic phases were determined. Accurate values of the phase transition temperatures for stoichiometric and  $\delta$ -spinels were found. The change in the elementary cell volume was found to be less than 0.5%, distortion which should not influence the working parameters of battery electrodes. Appearance of electric polarization and decrease of the dc conductivity was explained by ordering of electronic charges between  $\text{Mn}^{3+}$  and  $\text{Mn}^{4+}$  in distorted spinel.

[1] Lisovytskiy D., Kaszukur Z., Baumer N.V., Pielaszek J., Molenda M., Dziembaj R., Marzec J., Molenda J., Dygas J.R., Krok F., Kopec M., *Molecular Physics Reports*, 2002, **35**, 26-30.

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