Doctoral thesis abstract

"Physical Investigations of Nanomaterials for Lithium-ion Batteries"

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The aim of this thesis is a development of comprehensive and reliable methods for structural and optical characterizations of nanomaterials intended for the application as electrode materials in high performance lithium ion batteries. The study of such materials deserves a special attention due to the growing interest in these energy storage and conversion systems with regard to their possible stationary and mobile applications. It is noteworthy, that the materials discussed here and studied as part of this thesis were synthesized by inexpensive and simple preparation methods.

In the first part of this thesis, the basics of physical and chemical description of reversible lithium-ion batteries and a short description of the most promising active materials for the positive and negative electrodes were presented. In this study several experimental techniques (described in Chapter 2) were used, including *ex situ* and *in situ* methods, which allowed for the observation of structural changes of the analyzed materials and linking these changes with materials electrochemical activity. The third section covered the description of olivine- and spinel-based nanomaterials used in this thesis. The synthesis of these materials was also described.

The experimental part included thorough *ex situ* analyses of the materials listed above linked to the investigation of the doping and surface modifications effects. In particular, the reason for a beneficial effect of surface modification on the stability of electrochemical cells was described. In order to study the effect of the intercalation process on the structure of analyzed materials, the experiments on the electrodes charged and discharged in the electrochemical cells, were carried out. The study demonstrated the importance of using fast and comprehensive Raman analysis to investigate the surface of the electrodes. A particular emphasis was placed on Raman mapping mode, which gave the opportunity to combine the results of structural analysis with the performance of electrochemical cells and thus provided means for effective search of the causes of capacity fading.

The following part discusses the analyses of *in situ* annealing measurements of spinels and olivine precursors. The importance of *in situ* methods for the optimization of lithium ion battery material synthesis has been demonstrated. The proposed experimental procedure demonstrated how the choice of synthesis conditions influences the electrochemical properties of the material. Moreover, the *in situ* TEM annealing study allowed the observation of the formation of a new composite based on cubic manganese oxide and 3D carbon structures with an interesting spherical morphology. The mechanism of carbonaceous nanospheres formation was proposed and furthermore, it was demonstrated that starting from the same precursor, by manipulating only the annealing atmosphere, one can create of either anode or cathode materials with the same morphology. The possibility of a similar nanocomposite synthesis with other metal oxides or metals was discussed, in addition.

In conclusion to this thesis, experimental techniques were compared and advances in examination of material synthesis process and final optimization of lithium-ion batteries were summarized.