Measurement and Analysis of *in operando / situ* Lithium-Ion battery data on a XRPD laboratory diffractometer

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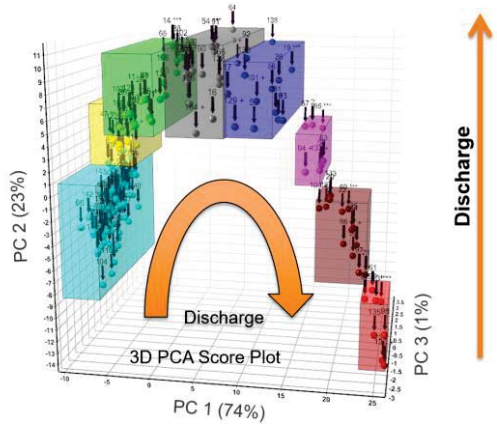
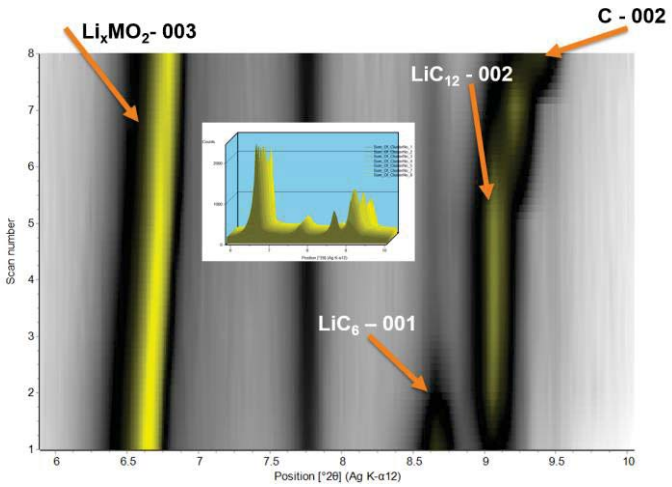
Typically, *in operando / situ* X-ray diffraction experiments of Lithium-Ion batteries are carried out on synchrotron beam lines due to the high brilliance sources and the possibility to use highly penetrating high-energy radiation. In this presentation however, we will cover how to quickly collect high (Rietveld) quality XRPD data of Lithium-Ion batteries during charge/discharge cycles on a laboratory XRPD diffractometer, equipped with an X-ray tube with Silver anode and an area detector optimized for high energy X-rays.
Further, we will discuss how to extract accurate phase quantities as well as crystallographic information automatically from multiple scans by using complex fitting models consisting of Pawley phases to model the fixed components (Aluminum and Copper electrodes), Rietveld phases to model variable components like $Li_{1-x}CoO_2$, LiC_6, LiC_{12} + Carbon and Profile fit peaks to model the polymer separator. Here we also propose a method how to model split peaks due to double pouch bag Al-foil seals and to extract useful spatial information.
Additionally, we will address how Cluster Analysis can be used to group and pre-sort the huge amount of raw data that is generated during these experiments. Cluster analysis can also be used for example to sort all scans of one or many experiments along a virtual charge/discharge axis.



1) 3D PCA Score plot and Cluster Analysis results of all charge/discharge cycles.



2) Virtual charge/discharge axis – summed clusters.

Keywords: high-energy radiation, XRPD, cluster analysis