Operando X-ray Studies of Lithium and Sodium Ion Battery Electrode Materials

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Lithium-ion batteries (LIBs) are the power source of choice for portable devices and electric vehicles. The technology is increasingly used for large-scale stationary energy storage to balance supply and demand of intermittent renewable energy. Lithium and sodium have similar chemistries and sodium-ion batteries (SIBs) may provide cost advantages over LIBs. Huge research efforts are currently underway to develop suitable SIB materials.

Understanding the working and degradation mechanisms of electrode materials at the atomic scale is fundamental to optimize battery materials. The key to this is investigation *in situ* during operation (also called *operando*). For this purpose a fully operational set-up (electrochemical cells, sample changer and interfacing software) that enables combined quasi-simultaneous operando X-ray diffraction (XRD) and X-ray absorption (XAS such as XANES and EXAFS) measurements coupled with electrochemical characterization was developed. The combined approach of *operando* XRD and XAS enables deep insights into electrochemical reaction processes (e.g. insertion, alloying, conversion), structural stability (e.g. pulverization, amorphization) and voltage-composition profiles (single phase vs. multi-phase) along with details on the oxidation state and the local environment of the electrochemically redox-active species.

Combined XRD and XAS analysis of the high voltage Li insertion cathode material $LiMn_{1.5}Ni_{0.5}O_4$, the high capacity Bi alloying and Bi_2S_3 conversion SIB anode materials, respectively, are provided and give deep insights into the working and degradation mechanisms of these electrode materials. The cells can further be used for total scattering (pair distribution function, PDF) and small angle X-ray scattering (SAXS).

The electrochemical cells and the setup are available at the Swiss Norwegian Beamlines at the European Synchrotron but can in principle be implemented at any synchrotron beam line. The cells can also be used on home laboratory diffractometers as we also demonstrate.

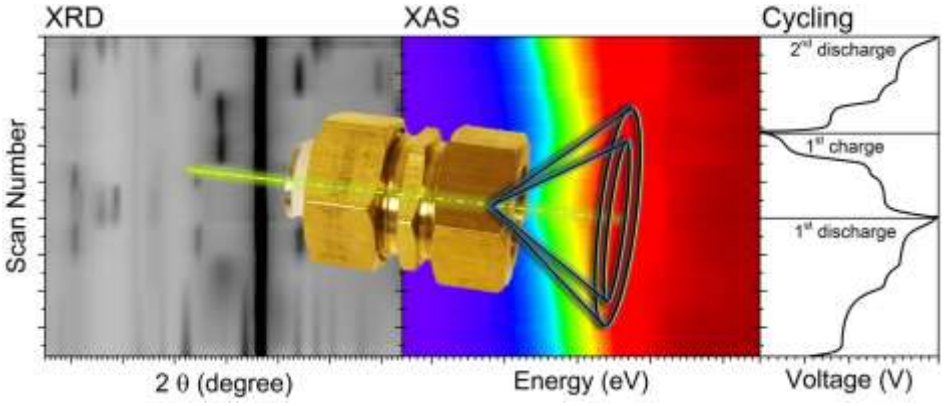

Figure 1 Illustration of *operando* electrochemical cell for non-aqueous batteries and its use for combined XRD/XAS measurements coupled with electrochemical characterization