influence for the design and operation of Li-ion cells, i.e. cell geometry (tab positions), cell balancing or operating temperature.

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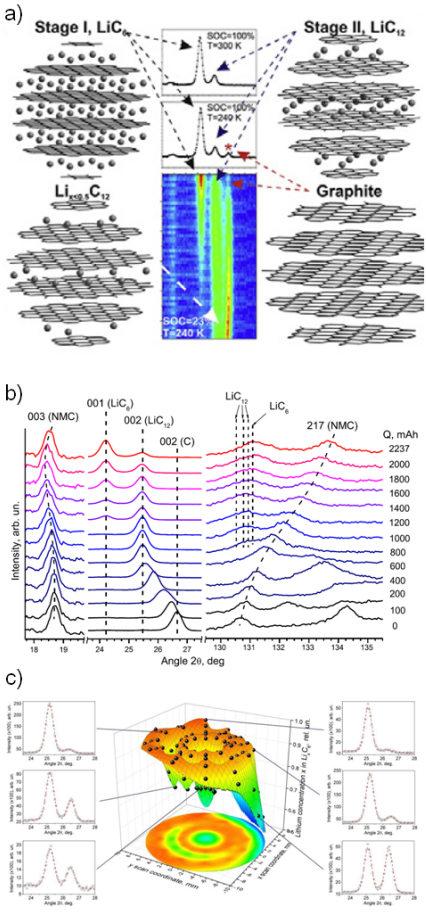

Figure 1. a) In operando neutron diffraction during discharge at 240 K, intercalation behaviour differs from room temperature [1]; b) diffraction data at different state of charge point out cation mixing in NMC cell [2]; c) spatially resolved lithium concentration inside the anode of a charged Li-ion cell [3]

Keywords: Li-Ion Battery, Neutron, Scattering, Diffraction

MS43-O2 New insight on structural and redox processes involved upon cycling of $Na_3V_2(PO_4)_2F_3$, an attractive positive electrode material for Na-ion batteries

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As electrode materials for batteries are operating in non-equilibrium conditions a deeper understanding of the structural and redox processes occurring upon cycling can be achieved using *operando* techniques. In order to do this a versatile electrochemical cell has been developed for X-ray experiments allowing real-time data collection upon charge/discharge (*i.e.* upon lithium or sodium extraction/insertion from/into the electrodes), either by lab X-ray and synchrotron X-ray powder diffraction or by X-ray absorption spectroscopy [1]. This unveils dynamics features that are not accessible by other means and gives a greater picture of the electrodes' functioning from electronic and atomic point of view.

Concerning $Na_3V_2(PO_4)_2F_3$ recent structural characterization of the fully charged material $Na_1V_2(PO_4)_2F_3$ *via* operando synchrotron X-ray diffraction performed at the ALBA synchrotron (Barcelona, Spain) suggests charge disproportionation of 2V(+IV) into V(+III) and V(+V). Indeed structural features combined with bond valence sums show two different vanadium environments consistent with such a hypothesis [2]. To further support this observation *operando* X-ray absorption near edge spectroscopy (XANES) at the vanadium K-edge in fluorescence mode has been performed at ALBA which gives relevant information on local environment and electronic configuration of the vanadium, especially through the pre-edge peaks' investigation. The obtained data have been analyzed *via* principal component analysis and multivariate curve resolution which constitute a novel approach to characterize electrode materials in operating conditions.

Furthermore impact of the cycling rate and temperature on the phase diagram observed upon sodium deintercalation and re-intercalation from/in $Na_3V_2(PO_4)_2F_3$ will be also extensively discussed.

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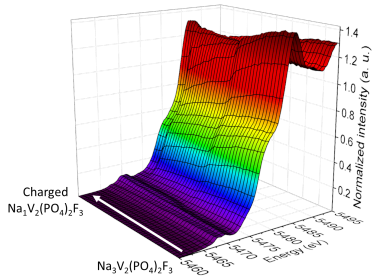

Figure 1. Vanadium K-edge XANES raw data collected operando upon charge of the battery Na//$Na_3V_2(PO_4)_2F_3$

Keywords: Na-ion Battery, operando synchrotron X-ray powder diffraction, operando XANES, NVPF

MS43-O3 In situ analysis of mechanochemical reactions using combined X-ray diffraction and Raman spectroscopy

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Mechanochemistry is increasingly used for synthesizing various materials including metal organic compounds and cocrystals.[1-3] Although this synthesis approach offers a fast and pure synthesis in high yields, there is a lack in understanding the mechanisms of milling reactions. The necessary data can only be obtained in *in situ* experiments, which were only recently established for milling reactions.[4,5] Herein, we present a novel setup enabling a combined in situ investigation of mechanochemical reactions using synchrotron XRD and Raman spectroscopy (see Fig.1). The specific combination allows to study milling processes comprehensively on the level of the molecular and crystalline structure and thus obtaining reliable data for mechanistic studies. Besides well-known MOFs like ZIF-8, the formation process of new metal phosphonates [6] and model cocrystals [7] could be studied in detail. The syntheses pathway of the different compounds could be revealed. The results prove that the presented method combination is applicable for a wide range of materials and will provide the necessary understanding to tune and optimize mechanochemically synthesized compounds.

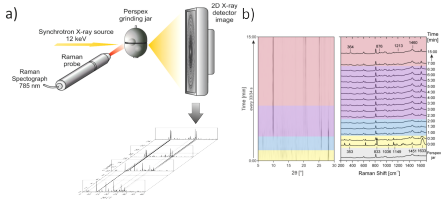

Figure 1. a) Schematic diagram of the experimental setup for collecting Raman spectra and XRD powder patterns during the mechanochemical synthesis. b) Synthesis process of the metal organic framework $(H_2Im)[Bi(1,4\text{-bdc})_2]$ followed *in situ* by synchrotron XRD (left) and Raman spectroscopy (right).

Keywords: in situ, mechanochemistry, Raman spectroscopy