PUSHING THE LIMIT OF OPERANDO TECHNIQUES TO PROBE BATTERY MATERIALS

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The Li-ion chemistry is thus far the most advanced chemistry employed in battery technology. To date, Li-ion batteries dominate the market of the electronics and portables devices. However, in the field of electric and hybrid vehicles further improvements are required in terms of performance, safety, and cost. Advanced Li-ion batteries and the novel systems (Li-S and Na-ion) utilize less explored electroactive materials and thus show new reaction mechanisms during electrochemical cycling, the understanding of which requires new characterization tools and techniques.
Development of a reliable electrochemical cells is thus of a prime importance when studying battery materials in *operando* mode during cycling. This is never an easy task, since the design of such cells has to be adequate to the technique of a choice and meet all necessary requirements.
Herein we present different cell designs developed in our laboratory and used for *operando* studies. Having overcome many obstacles, our *operando* cells are able to sustain more than 100 cycles and simultaneously to perform structural studies such as X-ray and neutron diffraction. For the latter one, we also tested and adapted a new set-up called stroboscopic mode. It allows *operando* study of the batteries that are cycling at very high rates (e.g. 10C) with a neutron patterns collected each 1 s along 200 cycles and more.

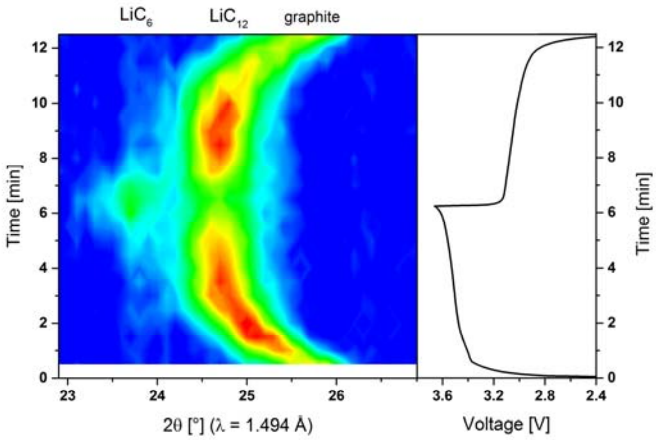

Figure 1. *Operando* neutron powder diffraction measurements using stroboscopic mode (cell cycled at roughly 10C rate) performed in a full cell $LiFePO_4$ vs. graphite (the plot shows the evolution of the main peak of the graphite (002) along charge and discharge).

Moreover, Li_2MnO_3 will be given as an example of the material that requires the activation at elevated temperature in order to be properly cycled. We modified our X-ray diffraction cell to be able to cycle it at temperatures higher than 25°C and thus to study the activation mechanisms of Li_2MnO_3. Other than that examples based on different *operando* techniques used to characterize Li-ion, Na-ion, and Li-S batteries will be also presented during the talk.

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