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Multi-technique analysis, correlation between ultrasound and x-ray diffraction on commercial cells

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Li-ion batteries currently dominate the electrochemical systems market but still suffer from ageing problems. Being able to determine the State of Health (SOH) of batteries is a critical need¹ to ensure user safety. Many studies on the ageing of battery materials have already been carried out, using multiple techniques, in particular using large scale facilities. These analyses allow *operando* monitoring of degradation mechanisms, but they are very long and difficult to set up²⁻³ and unfortunately cannot be used for use to monitor the day-to-day real-life conditions.

Ultrasound technology, which has been already widely used in many industrial sectors such as nuclear⁴ and medical⁵, has many advantages such as simple to implement, inexpensive, and enable non-destructive & *operando* measurements, allowing "*in vivo*" monitoring of the battery. However, it is difficult to know what an ultrasonic sensor can detect in a battery, whether it can determine the state of ageing of a battery and, above all, if the results obtained can be linked to any structural or morphological changes.

The objective of the study is therefore to correlate the analysis of ultrasound signals with X-ray diffraction measurements at the synchrotron (BM02 line at ESRF) in *operando* mode as a function of electrochemistry. For this purpose, a commercial cell composed of multiple electrodes of $\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$ (NMC 622) versus graphite (Z-stacking) was paired with an ultrasound sensor. X-ray diffractograms were acquired at several cycling rate and also during relaxation periods. The aim was to correlate the structural evolutions as a function of the charge/discharge rate and as a function of the ultrasound signal signature.

After checking if there is no beam damage on the sample, we were able to follow the evolution of the position of the two main reflections of the materials: the NMC (003) and the graphite (002) as a function of the lithiation rate (SOC) (Figure 1a).

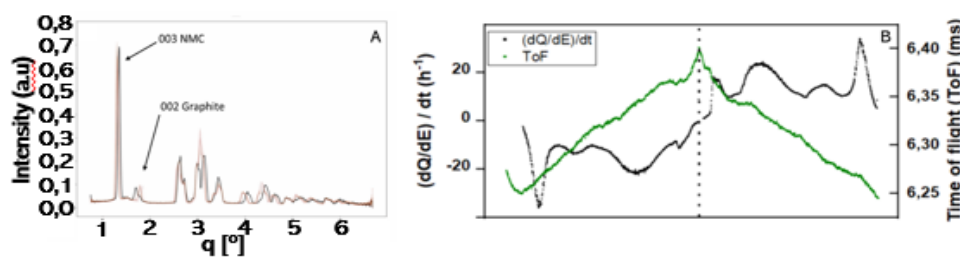


Figure 1. a) X-ray diffractogram acquired on the complete cell; b) Correlation between the electrochemical signal (derived from the galvanostatic curve) and the ultrasound signal

As can be seen in Figure 1b, we can correlate the electrochemical signal (derivative curve) to the change in the lattice parameters of graphite and NMC and to the time of flight (ToF) of the ultrasound showing that ultrasound can be an excellent non-destructive sensor to track structural changes within battery during cycling.

Through this talk, we will be presenting the methodology employed to correlate the results between XRD and ultrasound, as well as the additional techniques used to determine ageing parameters (impedance, X-ray tomography, dilatometer measurement, etc...).

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