

Experiment CH-4771: Investigation of phase transitions occurring in a layered oxide used as positive electrode for sodium ion batteries using operando diffraction

The experiment at ESRF was planned from August the 23rd to August the 30th on beamline BM32. The objective was to follow the structural evolution (strain, phase transitions) of a layered oxide electrode during the cycling of a sodium ion battery. Indeed, some of the structural transitions occurring during the successive charges and discharges of the battery are expected to be detrimental for its cycling stability. It is therefore essential to correlate the structure of the oxide electrode to the capacity of the sodium ion battery over several cycles. In other words, this experiment should establish the relationship between the sodiation/desodiation mechanisms and the structure of the electrode, an essential point to guarantee the reliability of the sodium ion batteries.

The results of these experiments have not been exploited yet but we expect that 50 % of the collected data can bring answers to the objectives described above. During our experiment, two electrochemical cells have been tested to analyze two different layered oxides. For the first sample, the XRD data for one charge and one discharge has been collected (Fig. 1). For the second sample (another oxide), the XRD data of only one charge has been amassed. We had not enough time to continue the battery cycling. However we hope this experiment will help us to better understand structural changes during the cycling of a layered oxide and to compare the two oxides.

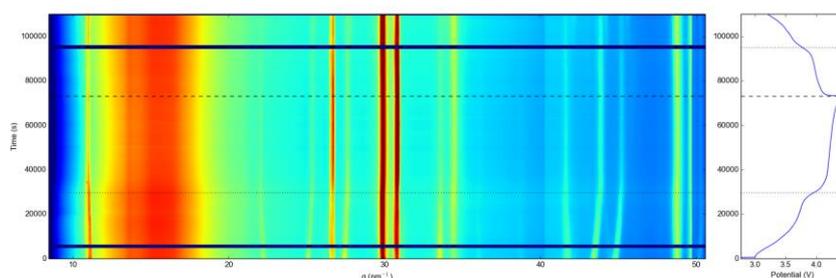


Figure 1. Preliminary results: (left) operando powder diffraction pattern acquired during the (almost complete) first charge-discharge cycle, (right) corresponding voltamogram. Analysis is in progress.

Thanks to the access to the electrochemical laboratory (EC-lab), we could take the cell to pieces, to clean it and to assemble another cell. All the materials we need were available in the EC-lab and the quality of the glovebox was very good. We are therefore much thankful to the staff of beamline ID03 and of the EC-lab for the access and the materials.

Unfortunately, beam failure has been a big issue during the shifts. During the electrochemical tests, a current is applied to charge or discharge the battery. When the current is stopped, the voltage of the battery decreases in charge (and increases in discharge) because of a self-discharge phenomenon, an intrinsic characteristic of main batteries. Consequently we didn't want to stop the current to avoid these self-discharge problems. Therefore, when the beam fails, the battery keeps cycling and the corresponding XRD data are lost. We experienced two beam losses: the first time the beam failure lasted less than 1hr so we lost 5% of the charge data. The second time, it lasted around 4 hrs (20%) of the discharge. As the second data lost was very important, we decided to assemble another battery to get the XRD spectra of, at least, one charge and one discharge.