ESRF	**Experiment title:** OPERANDO SAXS/WAXS TOMOGRAPHY ON MODEL AND COMPOSITE SILICON BASED-ANODES FOR LI-ION BATTERIES	**Experiment number**: **CH-5344**
Beamline: ID31	**Date of experiment**: from: 07 July 2018 (8h) to: 12 July 2018 (8h)	**Date of report**: 7 Sept 2020
Shifts: 18	**Local contact(s)**: Jakub Drnec	*Received at ESRF:*

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Report:

We developed a swagelock-type battery cell to perform the SAXS/WAXS tomography experiment on ID31 (shown in Figure 1a). The cell worked very well, loaded with NMC as cathode, standard liquid electrolytes and Si-based anodes. The experimental cycling sequence we could achieve is shown in

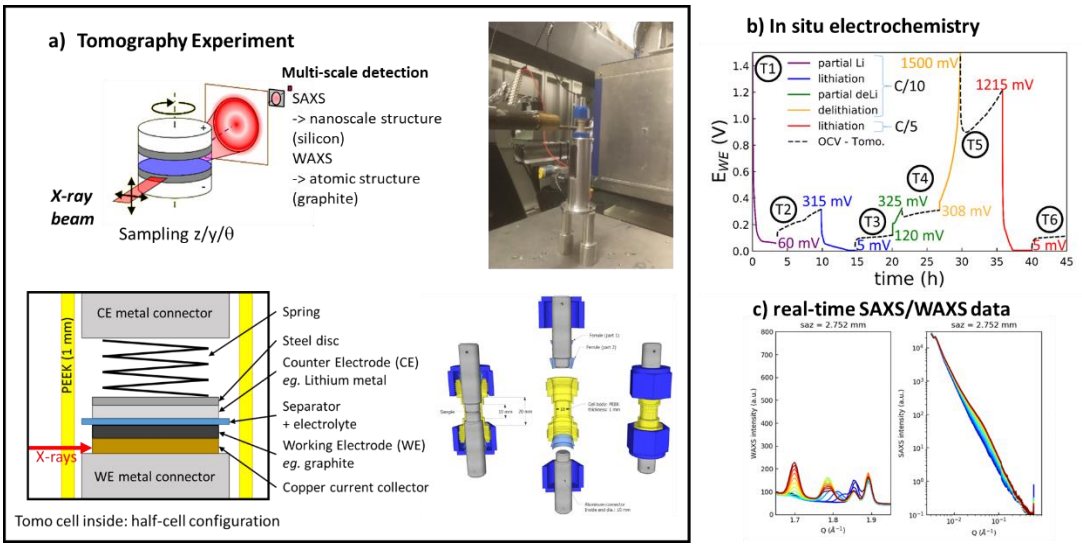

Figure b. We performed 6 tomography acquisitions at the potentials indicated in the figure. For each tomogram, we selected three vertical positions of the cell, e.g. we obtained 2D slices close to the separator, in the middle of the anode, and close to the current collector. The 2D reconstruction was performed by customizing the ID31 algorithm. Quantitative data analysis is on-going, but qualitatively the heterogeneous lithiation at the surface of the anode and in the three positions can be seen on Figure 2. It is seen that on the surface of the anode, the degree of lithiation of the graphite (given by WAXS data, left) and the silicon (given by SAXS data, right) depends on the localization both in-plane of the electrode and out-of-plane. There results highlight the great interest of this kind of measurements to probe in details the state of a silicon-

graphite composite anode during cycling. We are currently combining these results to modeling to better quantify the heterogeneities.

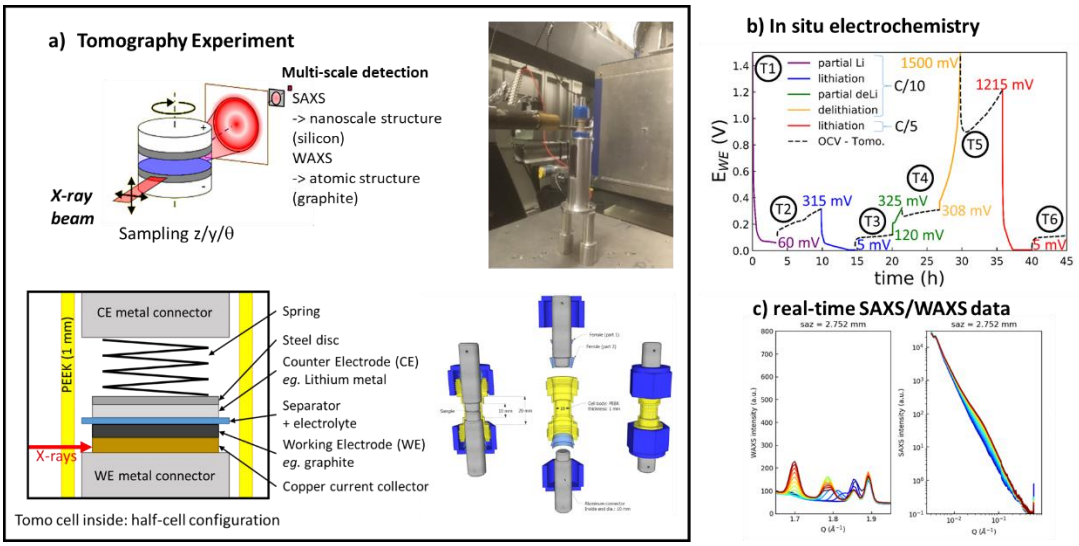

Figure 1. a) Principle of the synchrotron SAXS/WAXS tomography experiment and CEA tomography cell designed for it. Picture of the cell on ID31. b) Cycling sequence performed during the experiment and time where the 6 tomograms were taken (labeled Ti). c) Typical WAXS (left) and SAXS (right) data and their evolution in real-time during cycling at a given vertical position z.

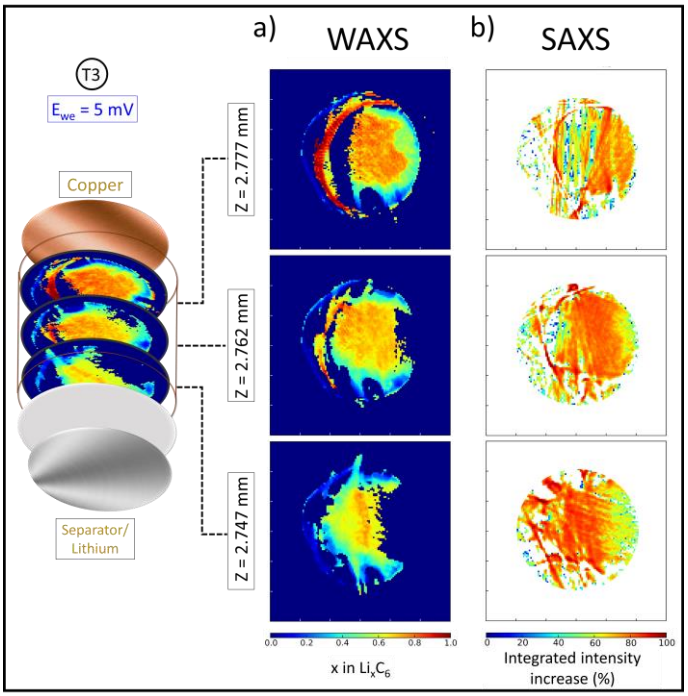

Figure 2. Analysis of WAXS and SAXS 2D patterns recorded at three different heights. From the WAXS we extract the degree of lithiation of graphite (e.g. x in LixC phases), and from the integration of the SAXS intensities we obtain some information on the degree of swelling (and, therefore, lithiation) of the silicon phase. As seen on the plots, the lithiation is heterogeneous in-plane, and also out-of-plane.