ESRF	**Experiment title:** Cycling and fatigue induced swelling of electrodes in 18650-type lithium-ion batteries	**Experiment number**: MA 5618
Beamline: ID11	**Date of experiment**: from: 10.02.2023 to: 13.02.2023	**Date of report**:
Shifts: 9	**Local contact(s)**: Pierre-olivier Autran	*Received at ESRF:*

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Report:

In this experiment a fresh commercial 18650-type lithium-ion battery was investigated using sub-µm operando CT at beamline ID11 in order to investigate the swelling behavior of the electrode materials.

In the first part of the experiment the cell was characterized at three different characteristic heights using an stitching approach with nine individual datasets. Beam energy was set to 88keV with an exposure time of 50ms for each of the 43200 projections with a pixel resolution of 0.65µm.

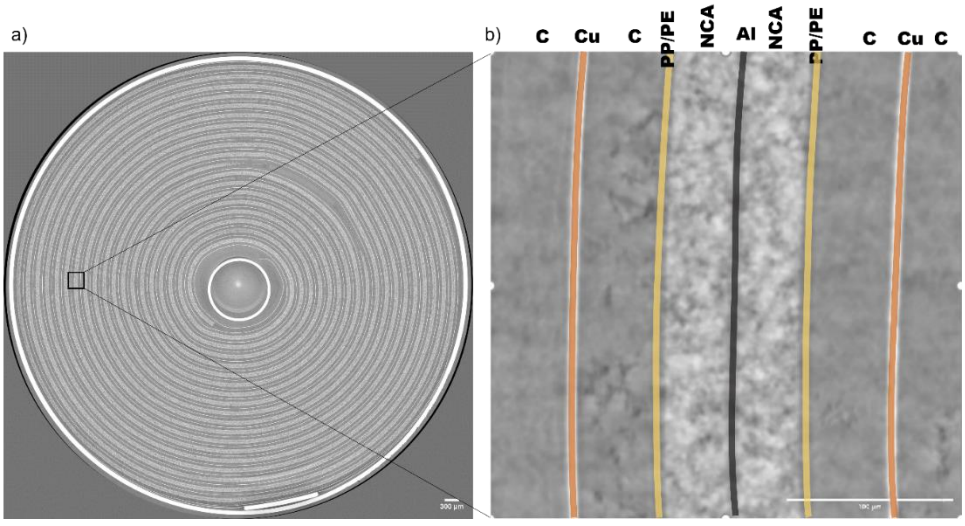

Figure 1 a) Exemplary slice through the investigated 18650-type lithium-ion battery and b) a zoomed in an arbitrary area.

Reconstruction of the data led to a full inside of the cell, where all individual cell components and their distribution were visible (Fig. 1a). Due to the high resolution of these CT pictures we are able to even observe

all individual particles of the electrode materials depleted on the current collectors (Fig. 1b). This approach reveals an overview about the distribution of the individual cell components and will be analyzed in detail.

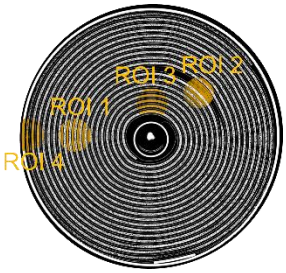

Figure 2 Location of the different regions of interest (ROI), where operando CT data was collected during dis-/charging of the battery

In the second part of this experiment the operando swelling behavior of the electrodes was investigated. Therefore, the cell was connected to a potentiostat and continuously electrochemically dis-/charged with a current rate of 2C/1C. Four regions of interest (ROI) were determined beforehand using the first part of the experiment. The localization of these ROIs is illustrated in Fig. 2.

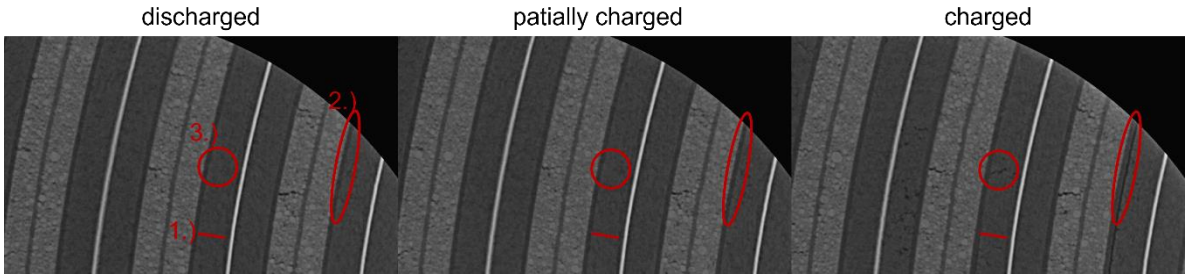

Figure 3 Reconstructed slices through ROI1 at different states-of-charge. Red marks indicate areas where 1) swelling/expansion 2) delamination of the electrodes and 3) electrode/particle cracking can be observed

In these regions CT data was continuously collected, while the cell was electrochemically dis-/charged. This approach led to the set of CT data, where the motion of both electrode slides and even individual electrode particles can be tracked during cell operation. Different interesting phenomena, where observed and will be evaluated in a more detailed way:

1.) Swelling/Expansion characteristics of the electrode slices itself (swelling/deformation)
2.) Delamination of the electrodes from the current collectors
3.) Electrode and particle cracking

All of these characteristics were observed in the CT-data as indicated in Fig. 3, where three different state-of-charge of ROI1 are displayed.
To further analyzed the data will be analyzed over the whole measured volume. In Fig.4 an exemplary 3D visualization of the copper current collector (small white stripe) and the NCA cathode (gray bars) are displayed.

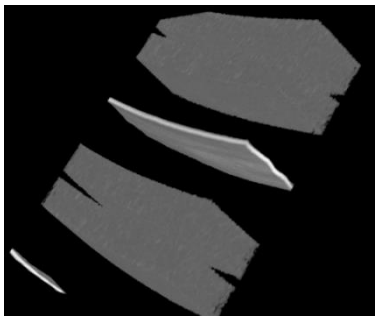

Figure 4 3D visualization of the current collector (white strip) and $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ cathode

At first glance the data quality seem to be good enough to determine the swelling behavior of the lithium-ion battery during dis-/charging. Additionally, in fresh cell unexpected delamination of the electrode material was observed along with particle cracking of the electrode material. Therefore, the aim of the proposal has been tackled a further informations about this topics have been collected.