	Experiment title: In situ PDF study of Li-rich layered oxide synthesis	**Experiment number**: CH-6713
Beamline: ID22	**Date of experiment**: from: 07/11/2023 to: 10/11/2023	**Date of report**:
Shifts: 9	**Local contact(s)**: Andy Fitch	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Background:
Li-rich layered oxides (LLOs) are promising cathode materials for Li-ion batteries that can deliver exceptional capacities due to combined cationic and anionic redox, but they still face substantial challenges related to cycling stability preventing commercialization. The origin is of a structural nature and it has been proposed that the key methodology to designing high-energy and long-cycle-life LLO-based materials is through structural engineering of the layer stacking sequence and tuning of local cation-cation interactions.

Aim:
In the present experiment, we intended to utilize the intensity and high resolution of the ID22 instrument to conduct in situ synchrotron total scattering (with pair distribution function analysis) studies of the evolution of the crystal- and local structures during the synthesis of LLOs. It is the hope that this will allow us to obtain fundamental insight into the chemistry and kinetic mechanisms at play as well as identify optimal reaction conditions and potential formation of metastable layered phases of interest.

People/team:
Present at the beamtime were Henrik Lyder Andersen (PI, MSCA Fellow, ICMM-CSIC), Matilde Saura (Talento Fellow, UCM) and Raul Artal (PhD student, ICMM-CSIC). The local contact was Andy Fitch who was assisted by Catherine Dejoie.

Instrumental configuration:
The instrument was set up with a beam energy of 60 keV (wavelength calibrated to 0.206689 Å from a LaB6 standard measurement) and data was collected using the Perkin Elmer 2D detector positioned at a distance of 380 mm (and initially switching to XRD mode with 1400 mm distance) behind the sample capillary giving a Qmax of approx. 25 $Å^{-1}$. The samples were heated using the retractable Cyberstar hot air blower (RT-950°C) mounted underneath the sample. The samples were generally heated at a ramp rate of 4 °C/min while dynamically collecting data at a time resolution of approx. 2.5 min (using tscan(50.1) command).

Technical problems:
Unfortunately, due to a technical issue related to the storage ring, the beam dropped in the afternoon of the 8/11/2023 and there was no beam at the ESRF until the end of the experiment (10/11/2023) resulting in the loss of approx. 6 shifts of beam.

Experiments/log:
After approximately 5 hours of setup time (incl. calibration and macro preparation), we started the first in situ experiment at around 13.15 on 7/11/2023. We managed to do 4 in situ measurements as well as a few room temperature snapshots (collected while cooling heater for next in situ measurements) before the beam went down. Figure 1 shows a representative dataset.

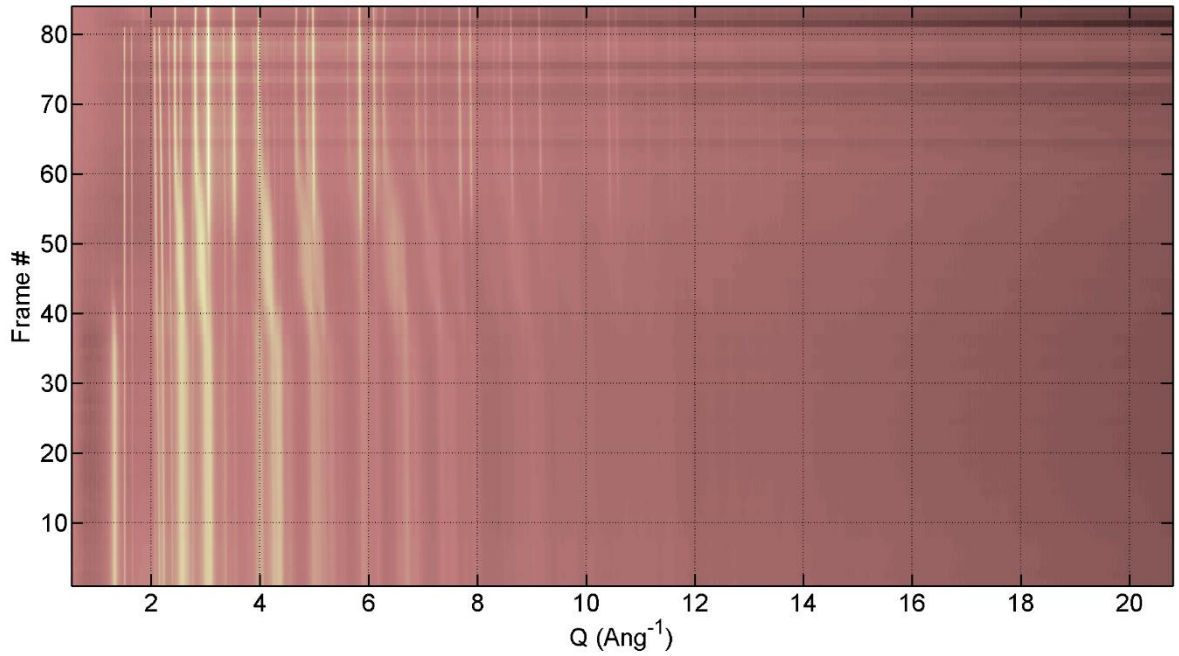

Figure 1: Background subtracted and intensity normalized in situ total scattering data collected while heating from RT to 950 °C on a pre-heated (2 h at 300 °C) sol-gel precursor.

Status:
Data analysis is ongoing and pending completion of the remaining 6 shifts of beamtime which has been tentatively scheduled in January 2024.