



Advanced Diffraction Methods for Characterization of Li-ion Battery Materials

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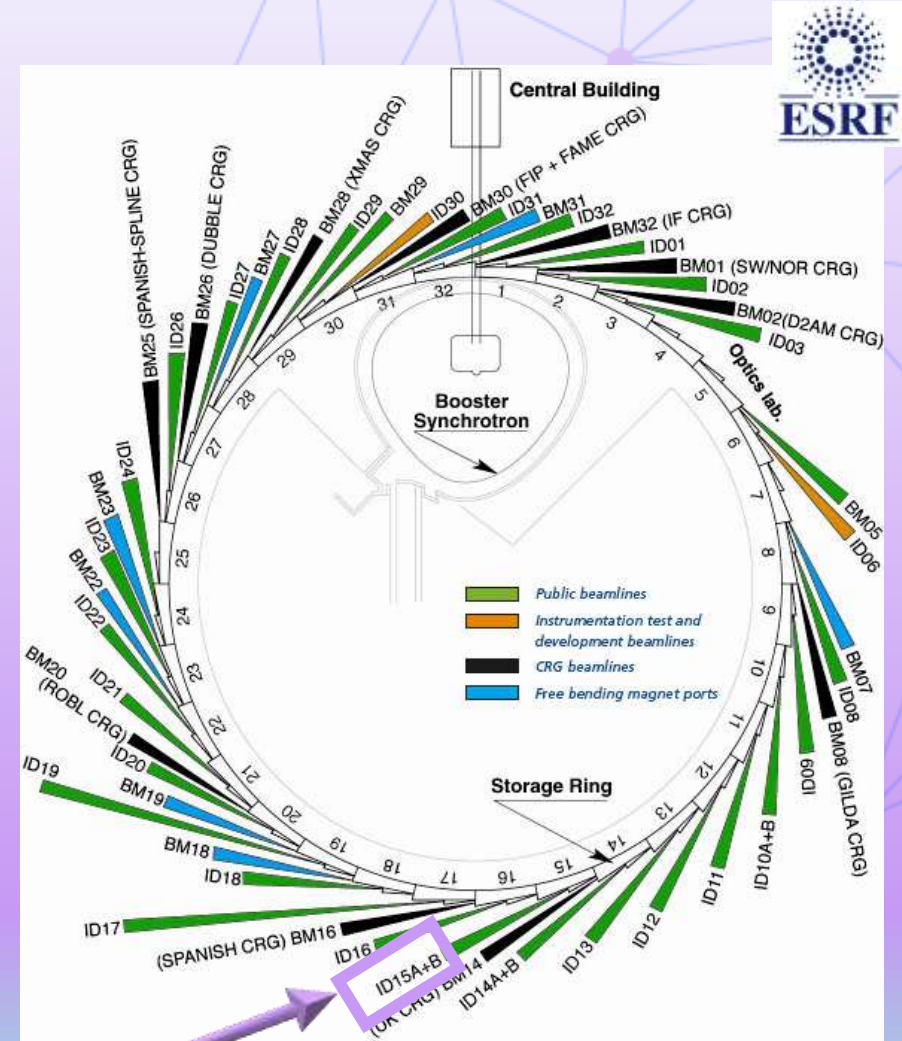
³ Johnson Matthey



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Who Am I?



ID15A Beamline

Beamline ID15A at the ESRF has been built and optimised for hierarchical **in situ/operando** studies of working systems, exploiting a variety of imaging, diffraction and spectroscopic techniques.

These techniques are particularly relevant to heterogeneous working systems such as **batteries** in which understanding the chemical and physical phenomena on several different length scales is critical to understanding the functioning of the system.

Beam size: 300 nm – 1mm

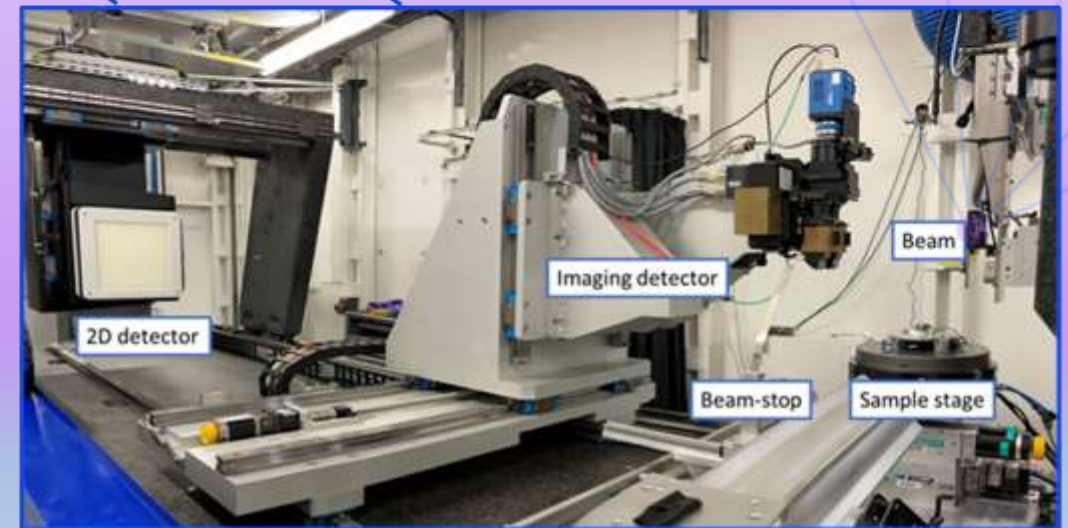
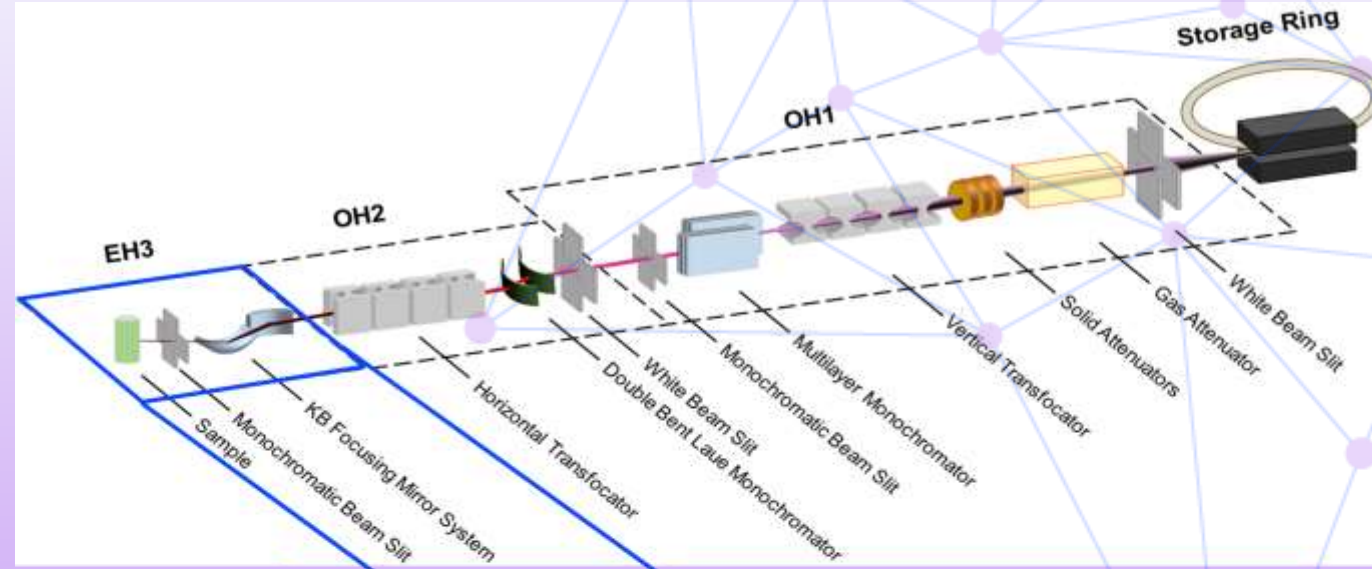
Energy: 20 – 140 keV

Bandwidth: 0.01 – 0.3 %

Different focusing modes available, sub- μm to 10s of μm s

Diffraction 2D detector: 20 bit dynamic range, frame rate 500 Hz, high efficiency up to 100 keV

Flux on sample increased with EBS: $\sim 2 \times 10^{13}$ ph/s @ 50 keV $\sim 3 \times 10^{12}$ ph/s @ 100 keV

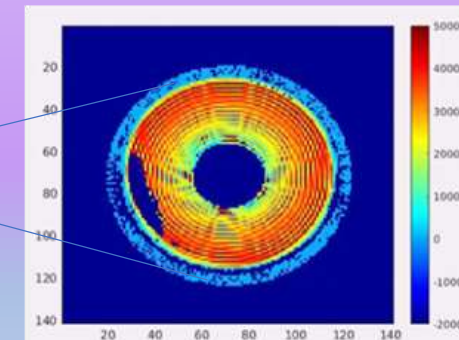
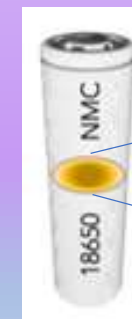
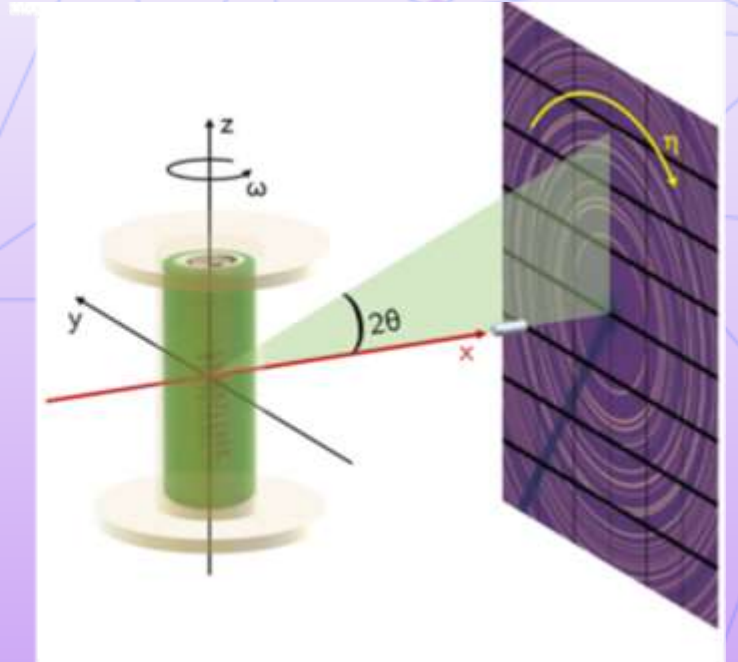


Project content and goals

At high-energy ID15A beamline at ESRF, 18650 cells were analyzed during cycling with x-ray diffraction computed tomography (XRD-CT) to obtain:

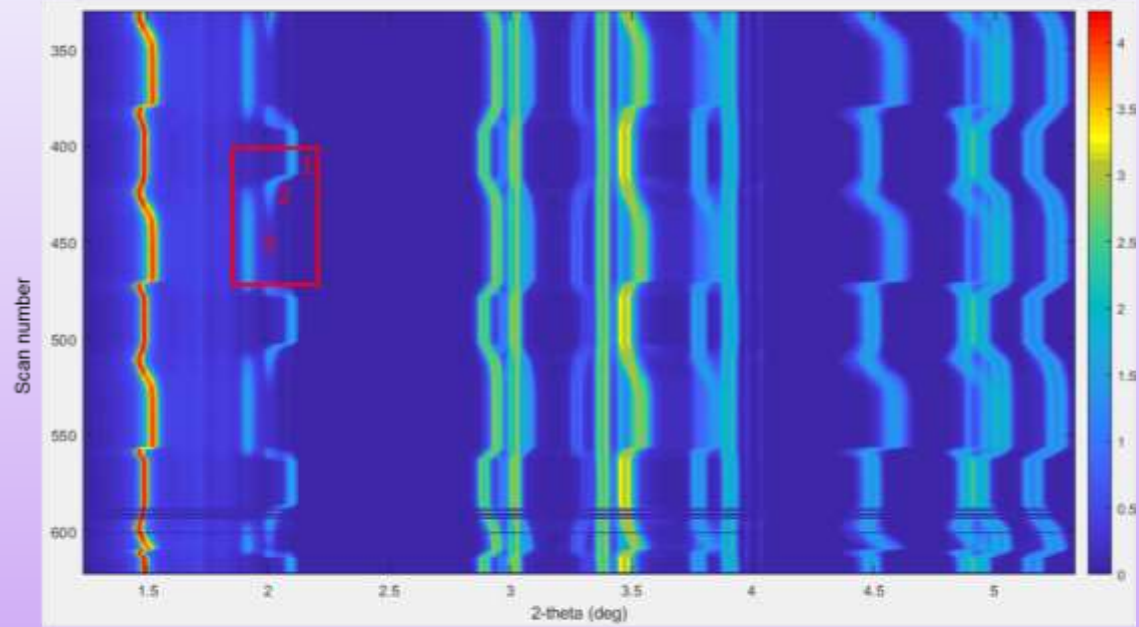
- Final reconstructed images evolving with time, in which each pixel contains diffraction information. Leading to electrode materials **phase transition map** during cycling.
- Evolution of Cu current collector lattice parameter for mapping **internal temperature variation** during cycling.

Working in partnership with **Johnson Matthey**, to translate these new methods to industry, to understand electrode material operation and degradation behaviour.

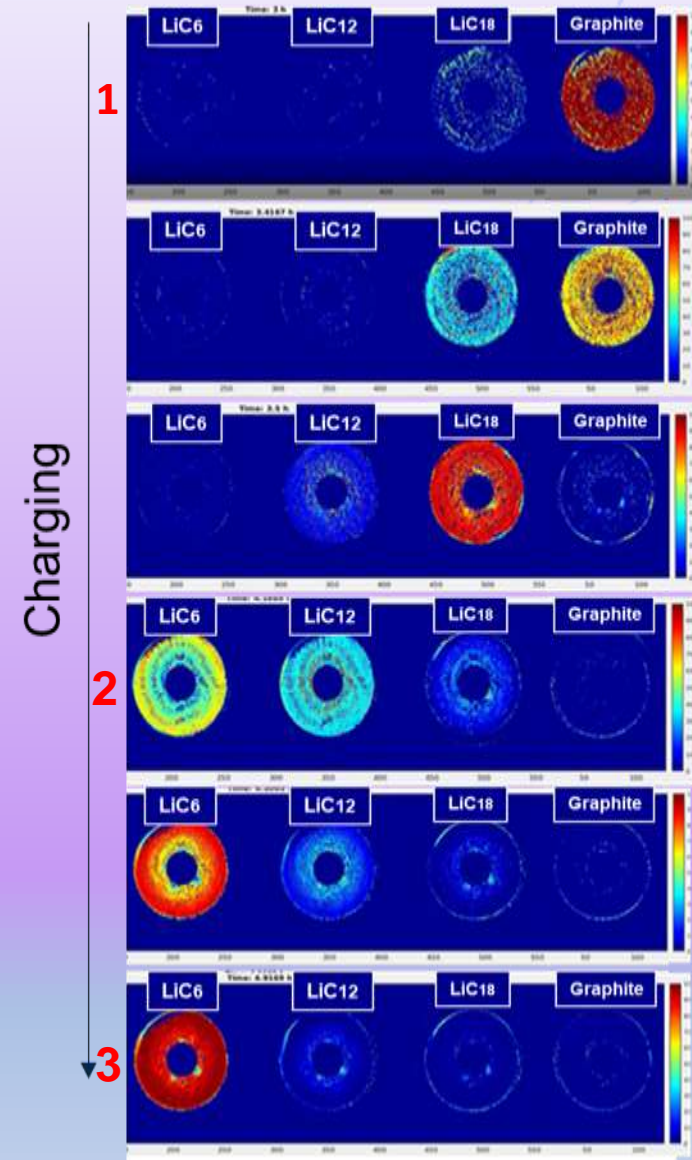


Operando experiments- Phase transition map

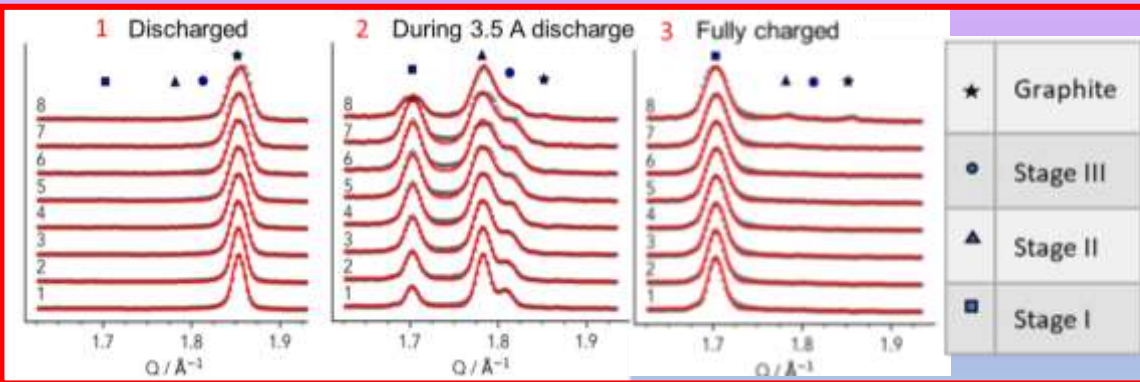
One point inside the volume during cycling:



Weight percent distribution of phases during cycling:

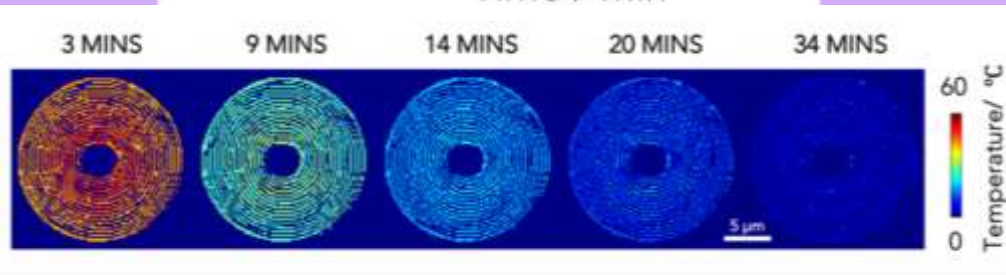
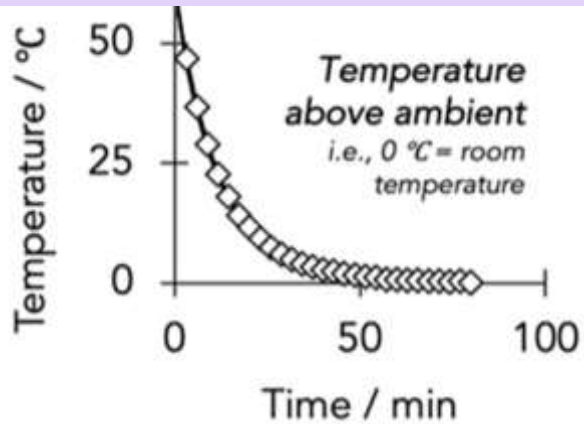


From diffractograms, final reconstructed images evolving with time contain in each voxel information such as crystal parameter or phase weight percentage of electrodes materials.

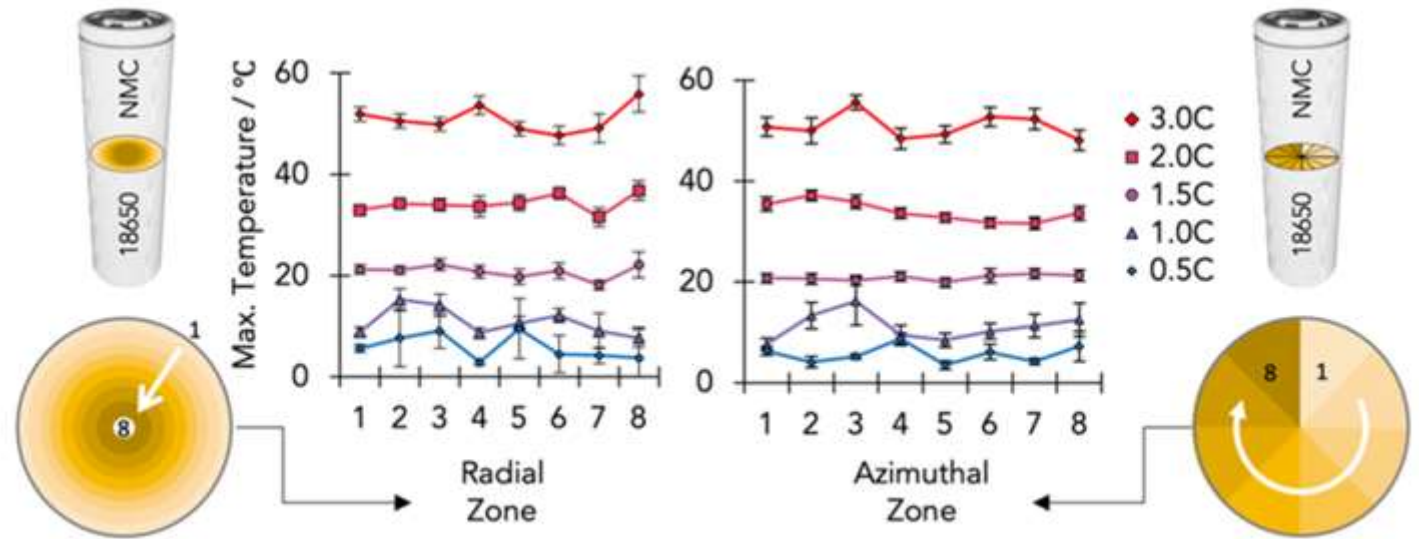
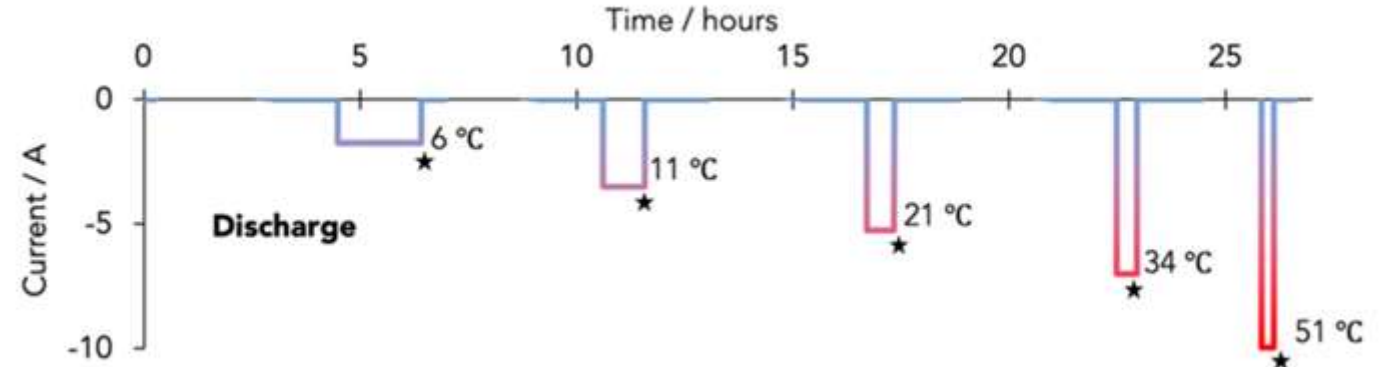


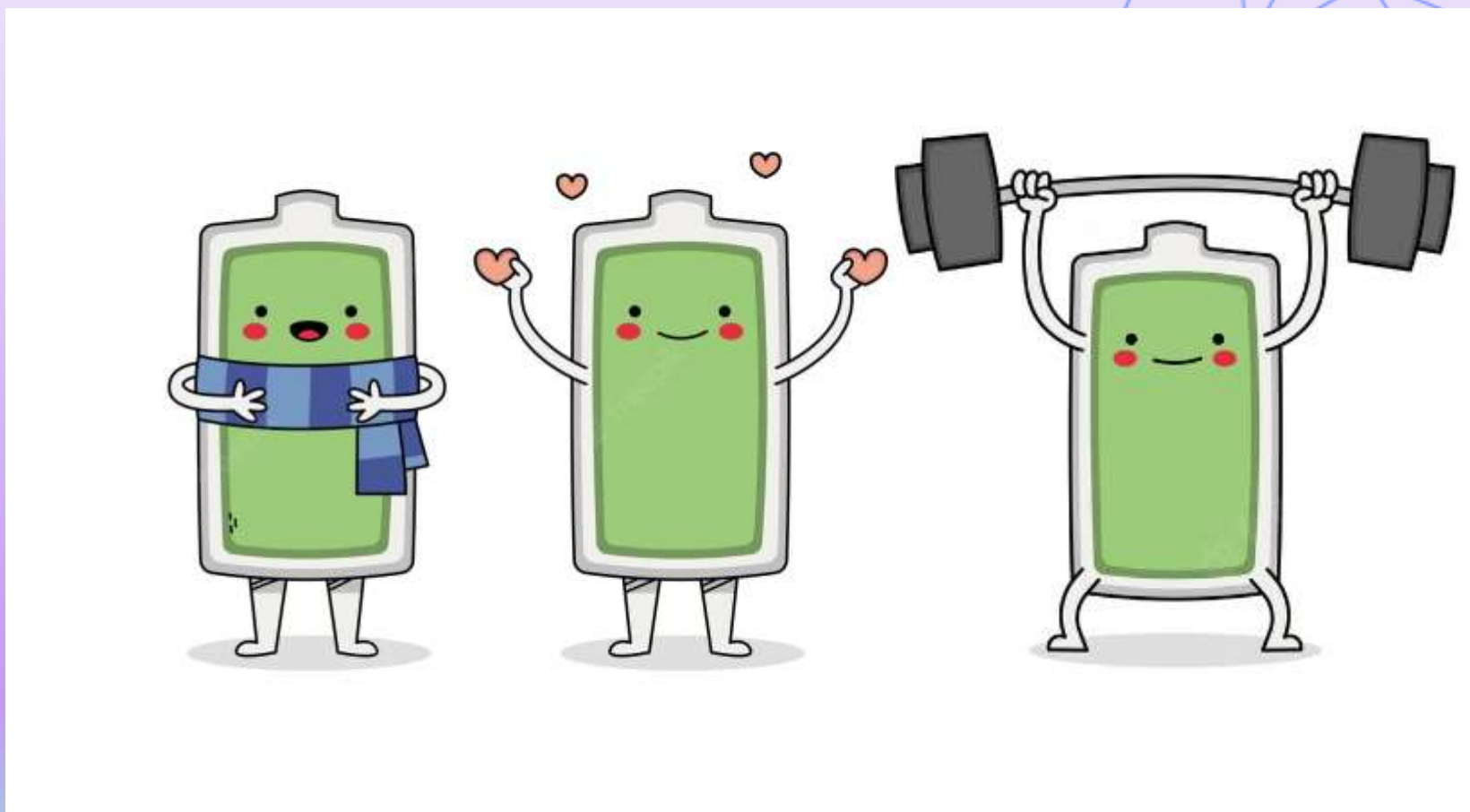
Operando experiments- Internal temperature detection

Temperature calculated from Cu current collector lattice thermal expansion
Cooling without electrochemistry applied:



During electrochemical cycling:





Thank you for the attention!