Hard X-ray Total Scattering- ESRF

TEESMAT
Open Innovation Test Bed for Electrochemical Energy Storage Materials

Principle

X-ray scattering or diffraction probes the ordering inside materials. With synchrotron beams, the structure of both crystalline and amorphous materials can be examined.

Why is it useful ? X-ray scattering provides quantitative information on the identity, quantity, and nanostructure of solid materials. This can be used to identify not only what solid materials are found inside materials, but also understand crystallite sizes, defect concentrations, strain, and other useful information on the structure of materials. Hard X-rays can penetrate through large samples (several cm), which allows full batteries to be examined without disassembly.

How it works

A microfocused X-ray beam illuminates a sample, and the elastically scattered X-rays are recorded. The beam size is ~2um, allowing very small samples or thin films to be examined. The high intensity of synchrotron X-rays allows high quality data to be collected in milliseconds, which allows diffraction imaging to be performed on cells, films, or other large samples.

What kind of sample ? Very few restrictions on sample geometry, mass, or composition.

Investigation time-scale : Very fast for simple ex situ samples, in situ experiments require more planning.

Maturity level : Well established

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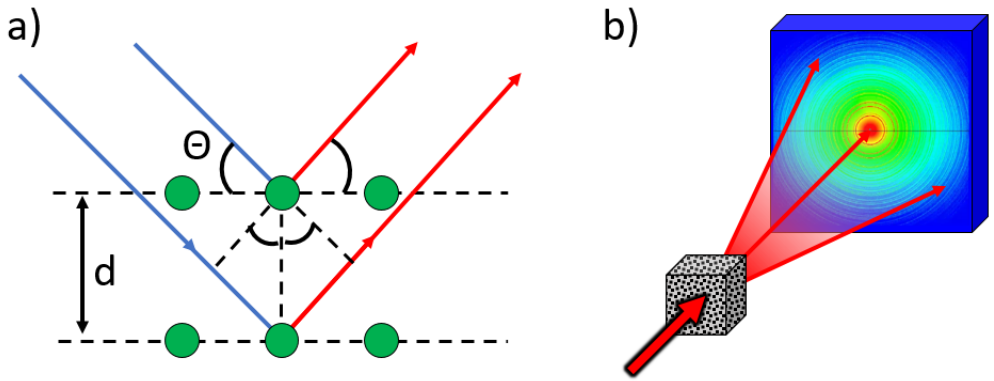

Figure 1. X-ray diffraction uses the interference between planes of atoms in crystals to reflect X-rays (a). Cartoon schematic of an XRD experiment, showing how X-rays are deflected by a sample, producing spots/rings on a detector.

Using a combination of small-angle X-ray scattering (SAXS), X-ray diffraction (XRD) and pair-distribution-function analysis (PDF), the structure of virtually all materials relevant for batteries can be examined. The information about the atomic structure often is the key to understand the processes leading to loss of activity and degradation and form the base for material development and assessment of stability.

Electrodes - Atomistic characterization and information about the fundamental processes during charge and discharge cycles, phase transitions and degradation.
Electrolytes - Following the changes in the structure of electrolytes during charge/discharge, aging of electrolytes.
SEI formation - development of SEI during conditioning of the cell.
Holistic studies - synergistic structural evolution between all materials of the cell in operando conditions.

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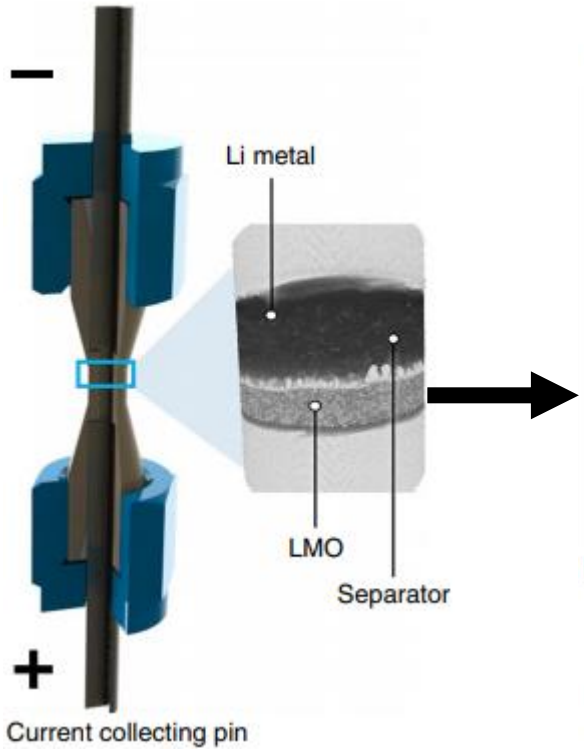
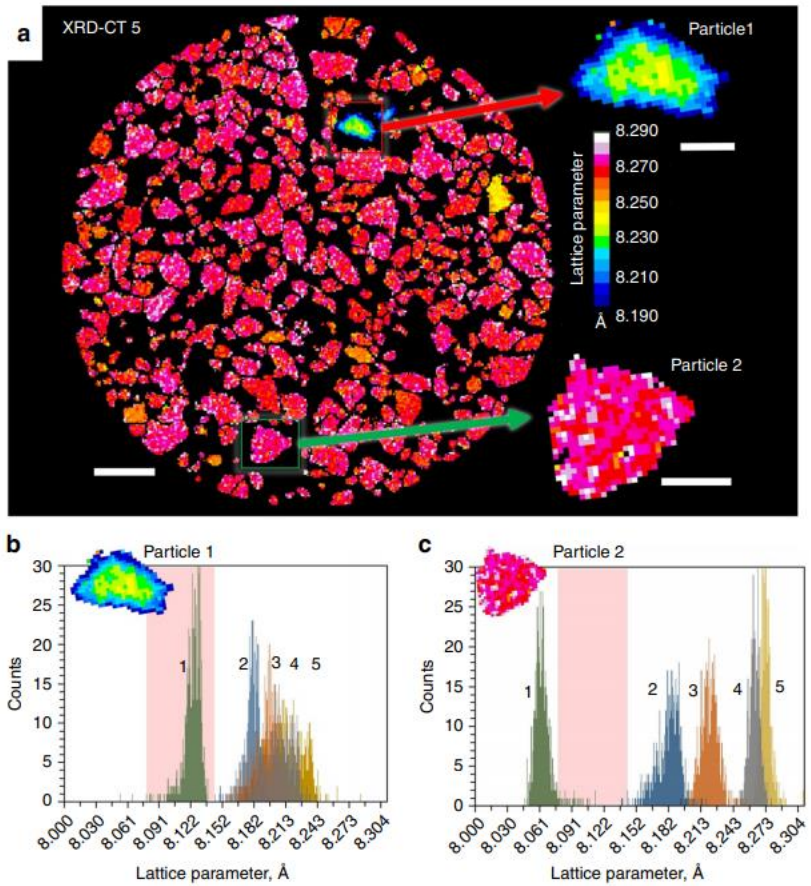


Illustration showing the design of the operando micro-cell with magnified X-ray CT reconstruction.



Full view of XRD computed tomography slice with two magnified particles of interest, one that significantly deviates from the bulk (Particle 1) and one that is representative of the bulk behavior (Particle 2). Scale bar for the bulk electrode is 40 µm and scale bars for enlarged particles are 10 µm. b–c Lattice parameter histograms taken from the individual particles at various states of charge (1–5). The pink region highlights the range of lattice parameter values that are not characteristic of pure spinel LixMn2O4. Taken from *Nature Communications,* **11**, 631, (2020)