X-ray Bragg diffraction microscopy - ESRF

TEESMAT
Open Innovation Test Bed for Electrochemical Energy Storage Materials

Principle

A nanofocused, X-ray beam scans across a sample, measuring the crystallinity and long-range order at the atomic scale by observing the diffraction pattern.

Why is it useful ? This technique is useful for nanoscale characterization of crystalline materials. Stress, strain, and orientation maps can be obtained with a resolution of approximately 30 nm. While electron microscopy offers higher spatial resolution, the capability of penetrating through relatively thick samples provides unparalleled *in situ* capabilities for battery investigations.

How it works

A synchrotron X-ray beam is rastered across a sample, and measures a single crystal X-ray diffractogram in each pixel, building up multidimensional images with strain and orientation contrast.

What kind of sample ? Highly crystallite solids, such as advanced cathode active materials. Must be radiation resistant. Electrochemical cell available.

Investigation time-scale : Typical imaging speed of 10-30 minutes per map, with single experiments of 1-5 days. Scheduling needs to be planned months in advance.

Maturity level : Extremely challenging, in development

X-ray Bragg Diffraction Microscopy - ESRF

TEESMAT
Open Innovation Test Bed for Electrochemical Energy Storage Materials

What can be seen

Imaging strain inside nanomaterials at the single particle level is critical for *in situ* mechanistic studies of intercalation, crystalline domain orientation, doping, and nanostructure. For example, Li (de)intercalation into battery active materials can be imaged with ~30 nm spatial resolution inside coin cells.

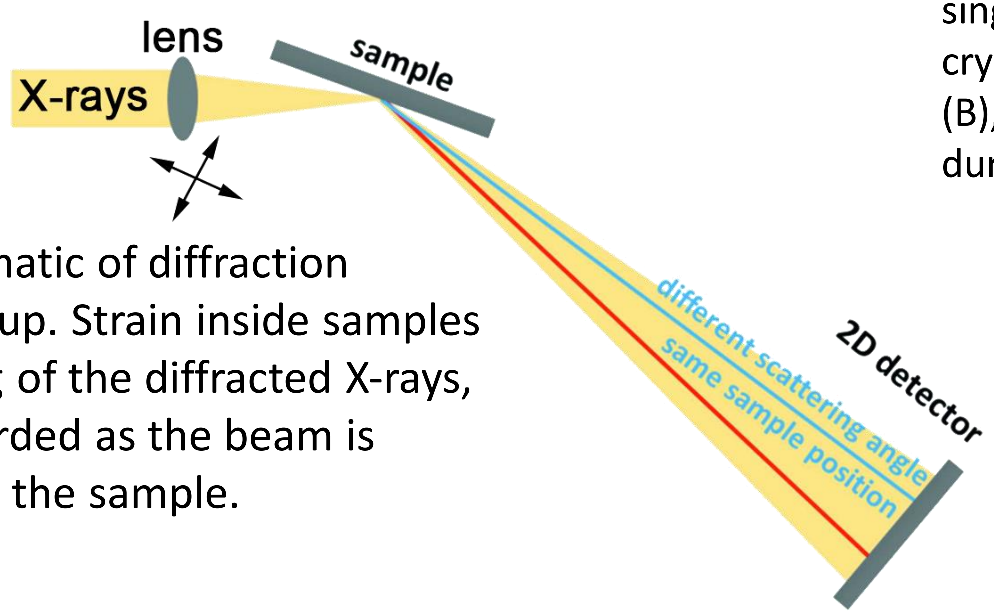

Figure 1. Schematic of diffraction microscopy setup. Strain inside samples causes bending of the diffracted X-rays, which are recorded as the beam is scanned across the sample.

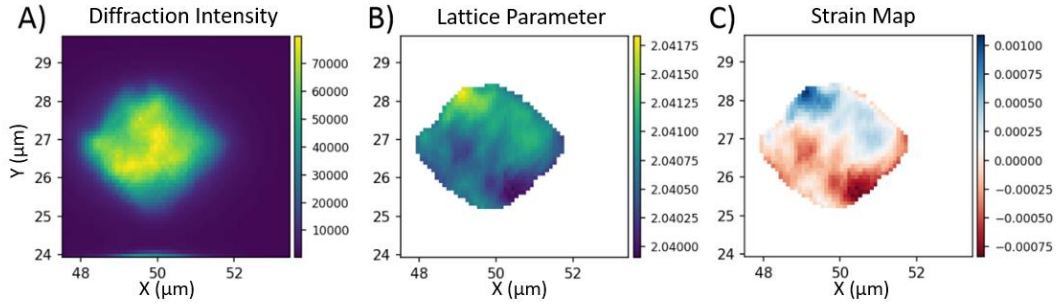

Figure 2. X-ray diffraction microscopy map of a partially lithiated single crystal of LMNO cathode active material (A). Variations in crystal structure of the active material can be measured in each pixel (B), allowing us to image the strain dynamics inside these materials during charging.