X-RAY DIFFRACTION

Battery CAM Application Package

Turnkey XRD methods for Li-ion battery cathode materials

The Battery CAM Application Package offers turnkey solutions for validating the structural quality and the phase purity of cathode active materials (CAM) used in lithium-ion battery technology. Based on X-ray diffraction, the industry-standard technique for crystallographic analysis, it enables automated, reproducible and accurate evaluation of key structural parameters that directly impact battery performance, including energy density, thermal stability, power capability, and cycle life.

By combining state-of-the-art XRD instrumentation, powerful analysis software, and Bruker's extensive application expertise, the package delivers push-button solutions for fast and reliable structural and microstructural characterization of CAM materials. This integrated approach minimizes user interaction while ensuring consistent and traceable results.

The package comprises four dedicated structural analysis methods designed to meet the requirements of process control across the entire CAM value chain—from precursor materials to finished active products. It is ideally suited for quality control in CAM production as well as for incoming goods inspection at battery manufacturing sites.

Figure 1 CAM powder.

pCAM turnkey solution – Stacking faults analysis in NMC precursors

Crystalline defects present in precursor materials (pCAM) may persist in the final active materials, negatively affecting their crystalline quality and their electro-chemical performance.

The pCAM method aims at assessing the crystalline quality in NMC precursors by quantifying stacking faults corresponding to specific combinations of rotational and translational mismatches between adjacent two-dimensional layers.

Based on the structural refinement of a brucite-type of $M(OH)_2$ layered hydroxide, stacking faults (both growth and deformation types) are introduced into a super-cell (10000-layer stacks) and quantified. Bruker expertise, combined with the flexible and powerful software DIFFRAC.TOPAS software, is driving the development of such innovative and unique solutions.

An optimized grid search algorithm reduces analysis times from hours to minutes compared to conventional stacking fault refinement approaches, making the pCAM method well suited for quality control applications.

Asynchronous data evaluation further increases sample throughput by allowing data acquisition and analysis to proceed independently.

Parameters that can be reported include:
- average lattice parameters
- growth and deformation fault
- crystallite size anisotropy
- degree of interstratification
- low-volume phase

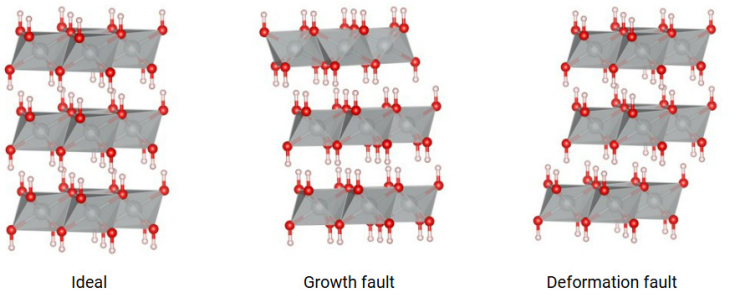
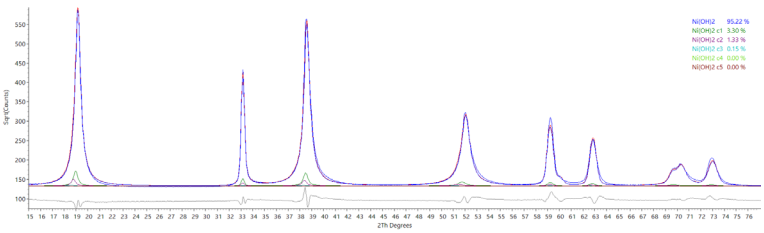


Figure 2 Ideal and defective layer stackings in NMC pCAM structure[1].



$(Ni_{0.9}Mn_{0.05}Co_{0.05})(OH)_2$

a (Å)	c (Å)	Growth faults	Deformation faults	Interstratification
3.1209	4.6270	8.5 %	0 %	5 %

Figure 3 Grid search stacking faults refinement with anisotropic crystallite size, isotropic strain, and interstratification.

CAM turnkey solutions

The Battery CAM application package includes three turnkey solutions to characterize the main cathode active materials (CAM) structure types used in Li-ion battery technology, namely LFP, NMC and LMO.

Each structure exhibits a distinct Li^+ diffusion mechanism—one-dimensional channels, two-dimensional pathways and a three-dimensional network, respectively—governing their power performance and rate capability. The application package delivers crystallographic insights correlated to the material properties.

Output parameters:
- Phase purity: secondary phases can be quantified
- Structural parameters: lattice parameters, atomic positions and site occupancies
- Microstructure: crystallite size and (an-)isotropic micro-strains.

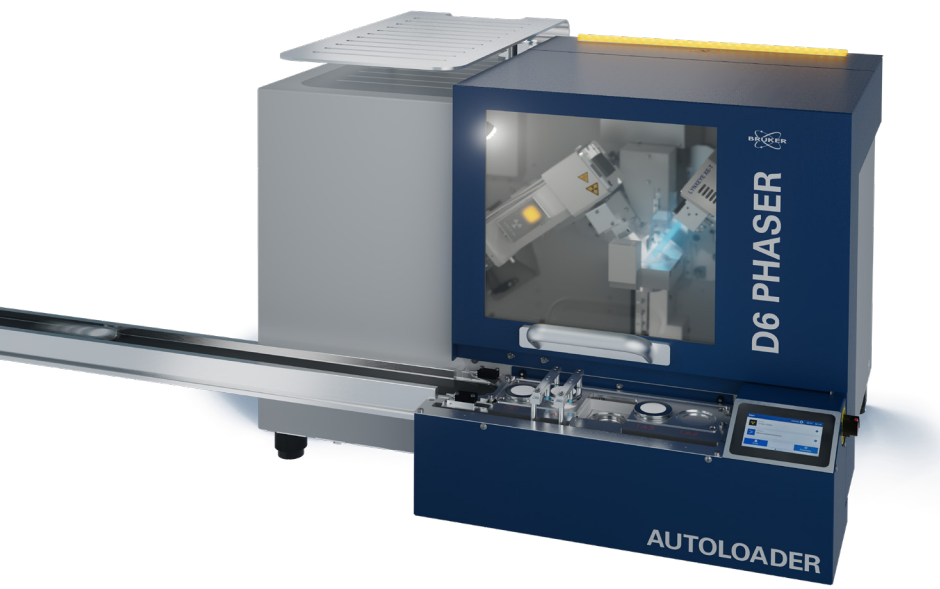


Figure 4 D6 PHASER AUTOLOADER benchtop XRD with connection to an automated sample preparation.

LFP turnkey solution

$LiFePO_4$ (LFP) adopts an olivine-type structure consisting of FeO_6 octahedra and PO_4 tetrahedra forming a robust three-dimensional framework with channels for Li-ion diffusion. The stiffness of this structure makes LFP a widely used cathode material in lithium-ion batteries, offering high thermal stability and excellent cycling durability.

The LFP turnkey solution applies to LFP-type materials, including compositional variants such as LMFP. A complete structural refinement provides all crystallographic parameters, including Li/Fe anti-site mixing, which particularly affect the one-dimensional Li^+ diffusion along the [010] channels.

NMC turnkey solution

$Li(TM)O_2$ oxides (NMC-type, TM = transition metal) adopt a layered structure exhibiting two-dimensional lithium-ion diffusion. This structural motif makes these materials prominent cathode candidates for high-energy applications due to their high ionic conductivity and excellent rate capability.

The NMC turnkey solution provides comprehensive structural and microstructural characterization for a broad range of cathode active materials (NMC, NMCA, NCA, and LCO). Key parameters driving Li-ion conductivity are c/a ratio, lithium slab thickness and degree of Li/Ni anti-site defects. While antisite defects impede Li^+ diffusion, they may also contribute to enhanced structural stability by mitigating lattice collapse during delithiation.

LMO turnkey solution

$LiMn_2O_4$ (LMO) has a highly-stable cubic spinel structure, providing a robust three-dimensional network for fast and isotropic Li-ion diffusion, making LMO a high-rate-capable cathode material for lithium-ion batteries.

The LMO turnkey method applies to LMO-type materials, including compositional variants such as LMNO.

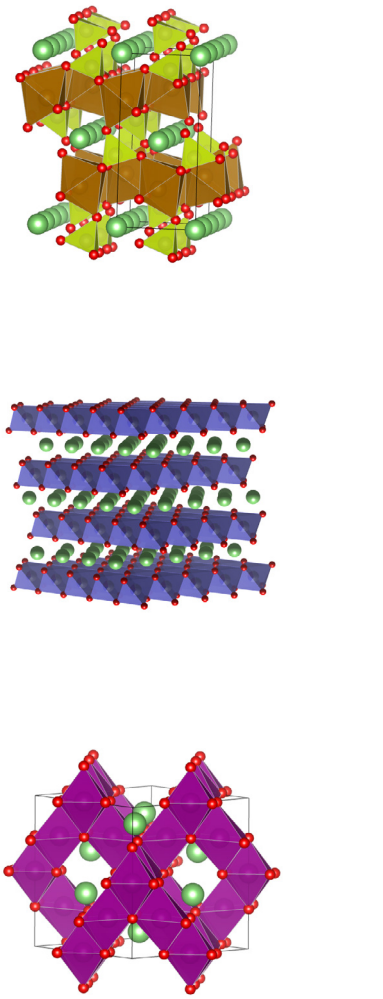
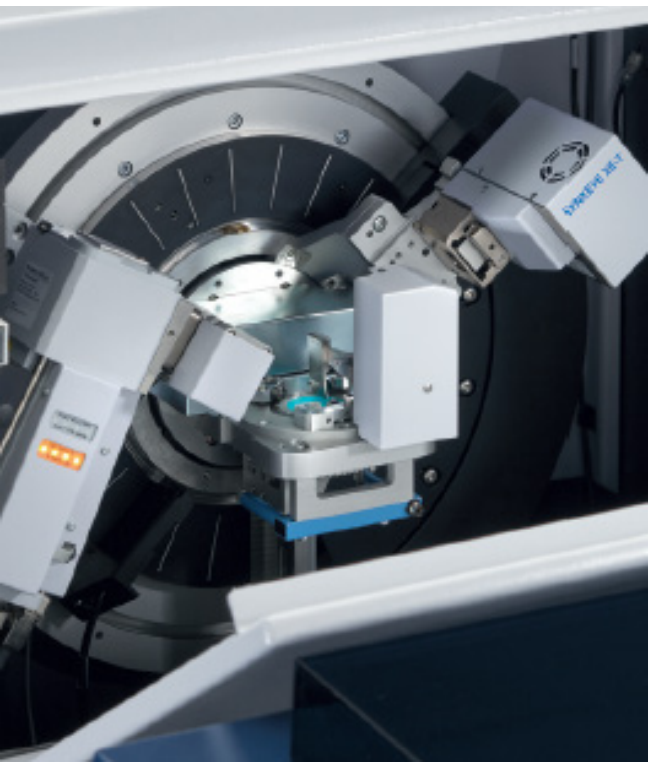


Figure 5 Structure representations of LFP-type (top), NMC-type (middle) and LMO-type (bottom) CAM materials[1].



State-of-the-art diffraction solution

Bruker AXS diffraction solutions uniquely support advanced beam-conditioning technologies, such as motorized air-scatter screens and motorized divergence slits.

These features enable Dynamic Beam Optimization (DBO), which automatically adjusts beam geometry and scatter reduction in real time. In combination with the energy-dispersive LYNXEYE XE-T detector—which filters fluorescence from transition metals—the resulting data quality is unmatched through optimal particle and counting statistics for accurate structure refinement and optimal signal-to-noise ratio for best limits of detection for secondary phases.

Figure 6 The goniometer of the D8 ENDEAVOR, the industry standard for high-throughput XRD quantitative analysis

DIFFRAC.TOPAS BBQ:
from expert-level analysis to push-button automation

DIFFRAC.TOPAS is the most powerful profile-fitting software for quantitative phase, microstructure, and crystal structure analysis. DIFFRAC.TOPAS BBQ translates expert-level Rietveld analysis into fully automated, production-ready workflows—enabling robust structural analysis directly into QC environment. The dynamic, sample-specific input of the transition-metal (TM) compositional ratio ensures reliable determination of structural parameters and a reduced number of recipes.

Seamless integration with laboratory automation systems and LIMS via the AXSCOM interface ensures efficient workflows and streamlined data management. This also ensures consistency in crystallite size evaluation across different instruments.

Consistent performance over time

To guarantee long-term reliability of microstructural parameters (crystallite size and micro-strains), the system continuously and automatically monitors the impact of X-ray tube aging on the instrumental contribution.

Technical Data

Evaluation parameters	Repeatability better or equal*	
Lattice parameters (CAM)	0.005% relative	
Lattice parameters (pCAM)	0.02% relative	
Crystallite size (< 300 nm)	2% relative	
Crystallite size (< 800 nm)	10% relative	
Strain e0	3% relative	
Site mixing	0.1% absolute	
Stacking faults probability (pCAM)	+/- 1% absolute	
Phase purity	0.3 wt.% absolute	
Sample throughput (includes calculation time)	2 samples/hour	D6 PHASER 600W, D8 ADVANCE, D8 ENDEAVOR ECO
	4 samples/hour	D6 PHASER 1200W, D8 ENDEAVOR

* St.dev. relative or absolute, minimum 10 repetitions on the same preparation
Conditions: Cu-Radiation, 4° Soller slits, LYNXEYE XE-T detector

References:

[1] K. Momma and F. Izumi, "VESTA 3 for three-dimensional visualization of crystal, volumetric and morphology data," J. Appl. Crystallogr., 44, 1272-1276 (2011).

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