XRD XRF XRM

Clean Energy

Applications of X-Ray Technologies along the Battery Life Cycle

Innovation with Integrity

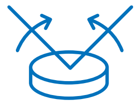
With advancements in battery technology, electricity has become an essential part of modern society, powering a wide array of devices, from smartphones and laptops to electric vehicles (EVs). As we navigate towards a low-carbon economy, storage systems will play a pivotal role in our energy evolution, storing renewable energy and reducing our dependence on fossil fuels. This has resulted in an ever-increasing demand for better batteries, both in terms of production capacity and performance.

However, the production of high-performance energy storage systems, such as Lithium-Ion Batteries (LIB) for EVs, relies on costly raw materials such as nickel, cobalt, and graphite. Coupled with the environmental impact of mining these materials, there is a growing emphasis on battery recycling, with governments even imposing recycling mandates.

As the industry scales up production and recycling efforts, there is a pressing need to enhance battery performance and reduce production costs. Achieving these goals means leveraging the right analytical tools.

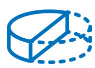
X-ray analytical techniques are indispensable along the entire battery life cycle. They facilitate the development of new cells and components, ensure quality control during manufacturing, and streamline the recycling process. These techniques are essential for optimizing battery performance, enhancing sustainability, and meeting the evolving demands of the energy landscape.

Overview of X-Ray Technologies



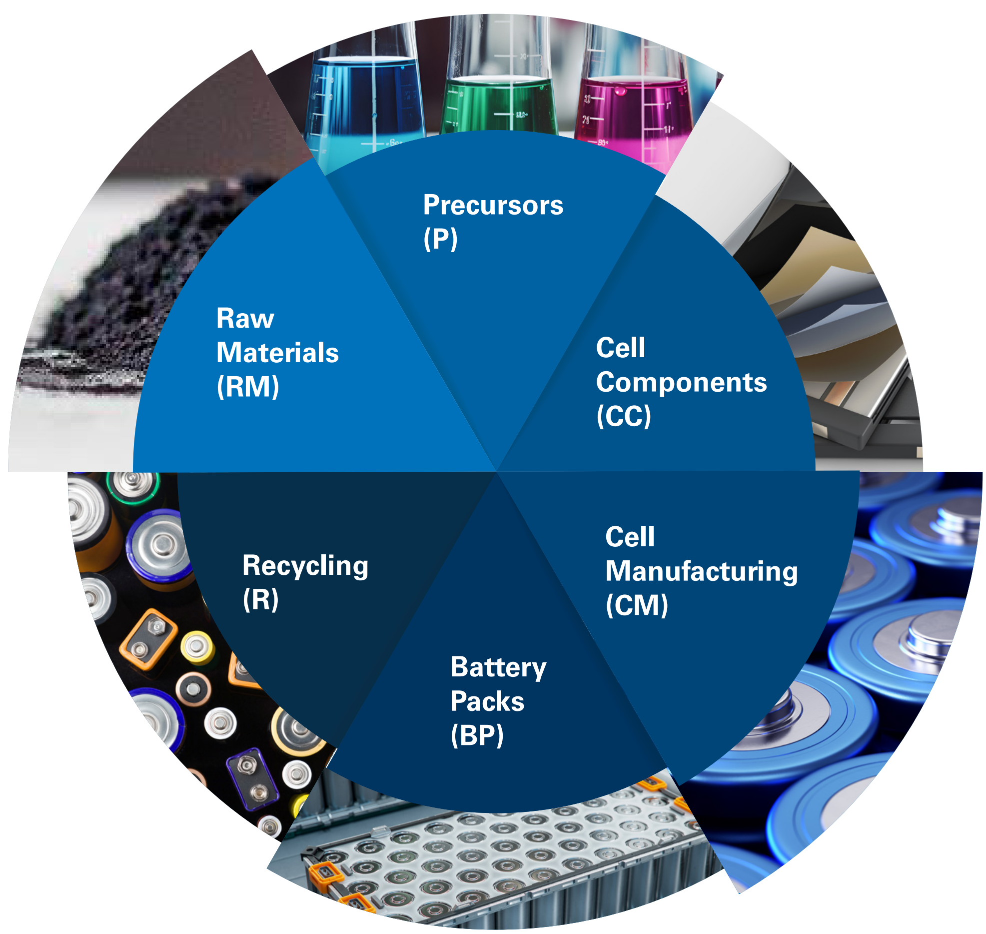
X-Ray Diffraction (XRD) provides quantitative structure information and allows for the analysis of dynamic changes occurring in a battery cell during cycling.

X-Ray Fluorescence (XRF) enables the determination of the elemental composition of raw materials, intermediates, and recycled black mass.



X-Ray Microscopy (XRM) provides a 3D internal view of precursor materials, battery cell components and assembled battery cells at the micro- and macroscopic level without the need for physical sectioning.

The Battery Life-Cycle



X-Ray Analytics for Raw Materials, Precursors, Cell Components

Raw Battery Materials and Chemicals

Several basic chemical and raw materials are required for battery cell production, which are typically retrieved from natural ore deposits or recycled batteries. X-ray technologies are being used for raw material identification (ID) and quality control (QC) of the following materials:

- **LiOH / Li_2CO_3:** Phase ID and purity (XRD)
- **Ni-, Mn-, Co-sulfate, Fe-phosphate:** ID and elemental purity (XRF), phase purity (XRD)
- **Graphite powder:** phase purity and polymorphism (XRD), contaminants (XRF)
- **Cu foil, Al foil:** Quality control (XRF)
- **Electrolyte:** Purity of conductive salts like $LiPF_6$ (XRF)

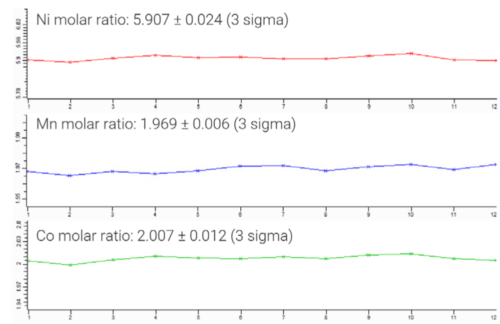


Figure 1
Ni-, Mn-, Co-molar ratio in liquid precursor determined by XRF.

Precursors and CAM production

Precursors include anything that has received further processing, like solutions and mixtures produced from sulfate salts. They also include intermediate products like hydroxide precursors and all steps involved until the final cathode active material (CAM) is ready. Common X-ray applications in this segment of the battery life cycle are:

- **Solutions / wet precursor:** Metal stoichiometry and purity (XRF)
- **Hydroxide-Precursor:** Metal stoichiometry and purity (XRF), calcination monitoring (XRD)
- **CAM powder:** Structure, lattice parameter refinement and crystallite size (XRD), particle size and shape (XRM), elemental analysis (XRF)
- **Solvent recovery:** Process monitoring based on recovered solvent composition (XRF)
- **Graphite:** Particle size and shape after spheroidization (XRM), foreign particle inspection (XRM)

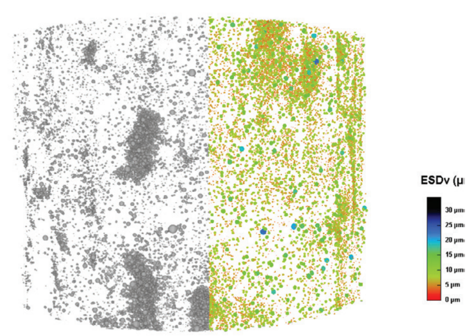


Figure 2
NMC cathode powder scanned with XRM at 800 nm resolution.Cathode particles partly color coded for size.

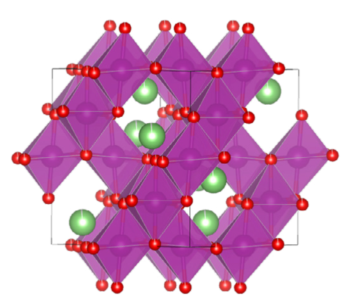
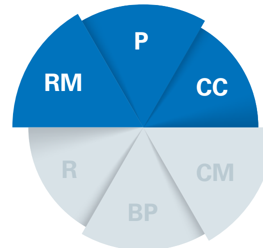


Figure 3
Crystal structure representation of LMO.



Cell Components

Cell Components are the building blocks that make up the battery cell, including coated CAM on aluminium electrodes and graphite on copper current collectors. X-ray applications include:

- **Cathode material:** particle size and distribution, tortuosity, void and microcracks, binder migration (XRM)
- **Anode material:** columnar growth in graphite (XRD, XRM), orientation index of graphite anode on Cu foil (XRD), SiOx volume and distribution in composite anodes (XRM)
- **Polymer separator:** Pore size distribution (XRM), analysis of property enhancing additives (XRF)

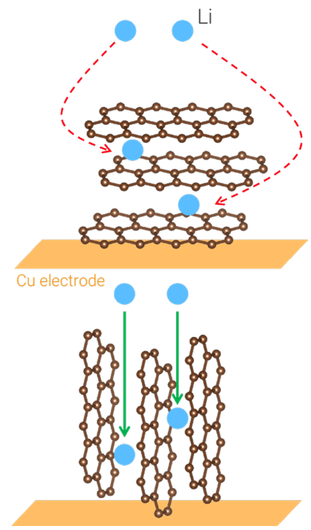


Figure 4
Orientation index determination of graphite on Cu foil using XRD can be used to qualify Li-ion conductivity in the anode.

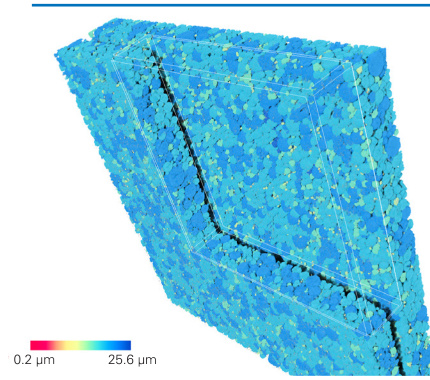


Figure 5
Quantitative analysis of CAM particle size with XRM, color coded for size.

X-Ray Analytics for Cell Manufacturing, Battery Packs, Recycling

Cell Manufacturing

Cells are the functional units of a battery. Depending on the final application they come in different formats: button, cylindrical, pouch or prismatic. X-ray applications include:

- **Operation:** operando measurements on pouch cells to track structural or phase changes in cathode or anode (XRD); gas formation (XRM)
- **Design inspection:** anode overhang, delamination, layer cracks (XRM)
- **Safety:** impact of nail puncture (XRM)

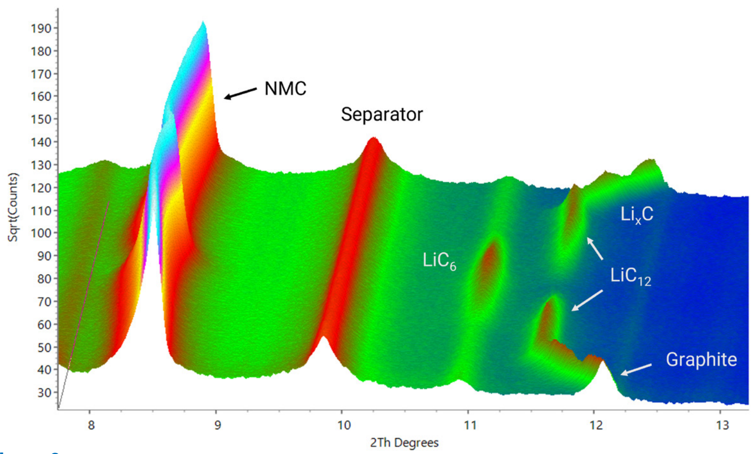


Figure 6
Surface plot of an operando XRD experiment on an NMC622 pouch cell shows the evolution of anode phases and structural changes to the cathode.

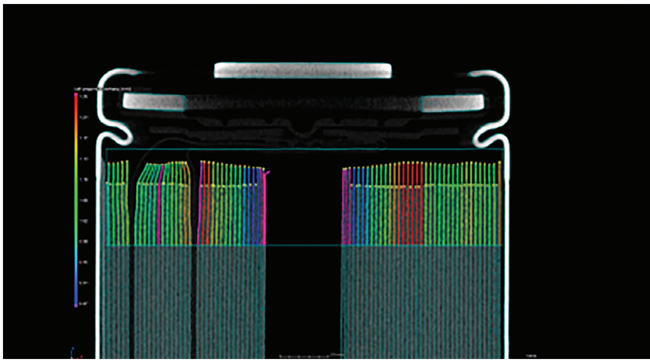


Figure 7
Anode overhang analysis in a cylindrical 21700 cell with XRM.

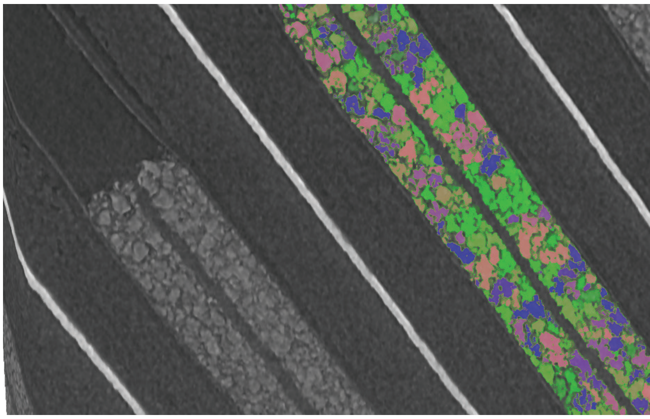
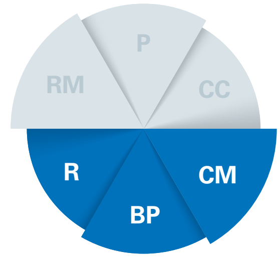


Figure 8
Lithium-ion pouch cell scanned with XRM at 500 nm resolution. Volume rendered 3D model with segmentation of the individual cathode particles.

Battery Pack Assembly

Individual battery cells get assembled into larger packs to achieve the power requirements, and combined with battery management systems for use in devices such as EV's, mobile phones, etc.

If an issue occurs during use, the packs are analyzed using a myriad of analytical techniques. Corrosion or combustion products are positively identified via XRF and XRD while the performance of internal safety mechanisms in the individual cells are checked using XRM.



Battery Recycling

Battery recycling is crucial in the battery life-cycle as it allows for the recovery of valuable materials like lithium, cobalt, and nickel, reducing the environmental impact of extraction while conserving resources.

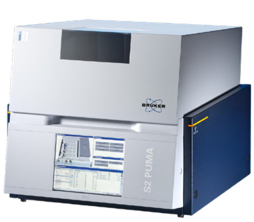
XRF has become an important tool for battery recycling companies. It is used to analyze battery black mass to:

- Determine metal contents (Ni, Co, Mn)
- Monitor contaminant levels (e.g., F, Al, Cu)

The XRF data is used for:

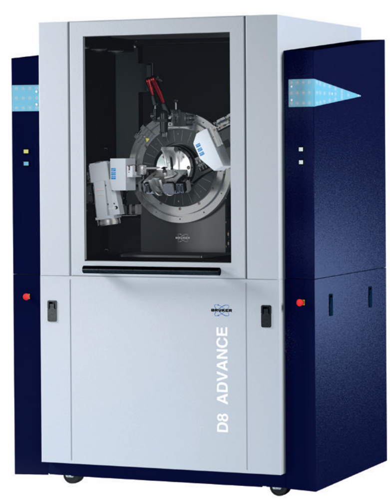
- Monitoring and optimization of mechanical separation
- Value determination (e.g., Ni content, seller & buyer)
- Quality control (e.g., impurities) to ensure a cost-efficient hydrometallurgical treatment of the black mass

Figure 9
Battery black mass powder.



S2 PUMA Series 2 (XRF)

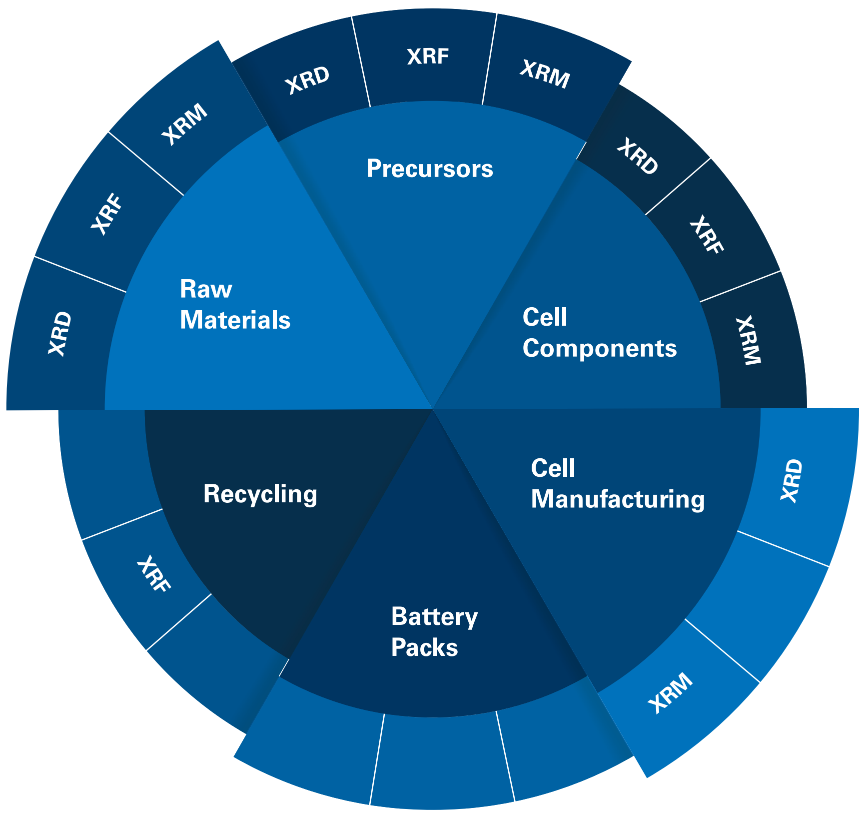
SKYSCAN 2214 CMOS Edition (XRM)



D8 ADVANCE (XRD)

Advantages of X-ray Technologies

- Non-destructive analysis to streamline product development and quality control (QC)
- Comprehensive characterization over different length scales: From crystal structure to 3D cell design
- Improve production KPI’s by linking manufacturing processes with quantitative analytics



Bruker AXS
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

bruker.com