IMPRESS SCHOOL
on *Operando and Correlative Experiments*

May 5-9, 2025
ALBA Synchrotron, Barcelona, Spain

Operando Characterization of Battery Materials

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The study of battery materials under real operating conditions is crucial for understanding their fundamental electrochemical mechanisms and improving performance. Operando synchrotron radiation-based characterization techniques have become essential tools for this purpose, enabling non-destructive probing of materials with varying depth sensitivities through spectroscopy, scattering, and imaging techniques.[1] Synchrotron-based techniques offer key advantages such as faster acquisition rates, tunable penetration depths, higher spectral and spatial resolution, and access to experimental approaches that require a continuously tunable source over a broad photon energy range. Ensuring compatibility between electrochemical cell designs and experimental setups is critical to preventing perturbations in the electrochemical response of the materials under investigation. Properly designed operando cells enable real-time tracking of structural, electronic, and chemical changes, capturing metastable intermediates and eliminating artifacts that can arise in ex situ studies due to sample evolution during handling.[2] Combining multiple synchrotron-based techniques further enhances the understanding of battery materials by providing a multidimensional perspective across time, space, and energy scales. This talk will present recent progress in operando synchrotron characterization of battery materials, focusing on X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS), and Fourier-transform infrared (FTIR) spectroscopy. Key aspects related to experimental setup, data acquisition, and data processing will be discussed,[3] along with challenges such as radiation-induced artifacts[4] and technical limitations. Practical examples will illustrate how these techniques contribute to advancing energy storage research, from fundamental battery chemistry to applications in emerging technologies.

1. Black, A. P. *et al.* Synchrotron radiation based operando characterization of battery materials. *Chem. Sci.* **14**, 1641–1665 (2023).
2. Choudhary, K. *et al.* Operando X-ray diffraction in transmission geometry « at home » from tape casted electrodes to all-solid-state battery. *J. Power Sources* **553**, 232270 (2023).
3. Arcelus, O. *et al.* FullProfAPP: a graphical user interface for the streamlined automation of powder diffraction data analysis. *J. Appl. Crystallogr.* **57**, 1676–1690 (2024).
4. Black, A. P. *et al.* Beam Effects in Synchrotron Radiation Operando Characterization of Battery Materials: X-Ray Diffraction and Absorption Study of LiNi0.33Mn0.33Co0.33O2 and LiFePO4 Electrodes. *Chem. Mater.* **36**, 5596–5610 (2024).