doi:10.2533/chimia.2026.413 Chimia 80 (2026) 413 © Swiss Chemical Society

Highlights of Analytical Sciences in Switzerland

Division of Analytical Sciences

A Division of the Swiss Chemical Society

Unveiling Secret Battery's Degradation and Functioning Mechanisms *In Situ* with *Operando* Synchrotron Chemical Imaging

Chayene G. Anchieta[a,b]*, Barthélémy Lelotte[c], Hari Vignesh Ramasamy[c], Alexander Forster[d], Ali Coskun[d], Mario El Kazzi[c], and Dario Ferreira Sanchez[a]

*Correspondence: C. G. Anchieta,
E-mail: chayene.anchieta@ntnu.no; chayeneanchieta@gmail.com
[a]Swiss Light Source, PSI, Villigen, Switzerland; [b]Department of Physics, Norwegian University of Science and Technology, Trondheim, Norway; [c]PSI Center for Energy and Environmental Sciences, PSI Villigen, Switzerland; [d]Department of Chemistry, University of Fribourg, Fribourg, Switzerland.

Keywords: All-solid-state battery · Chemical-imaging · Diffusion gradient · *Operando* XRD · Synchrotron

Understanding the degradation of batteries remains one of the most critical challenges in energy storage research. The success of Li-ion technology has been driven by continuous advances in materials and components, and further progress will depend on deeper insights into how materials evolve during operation. Tracking physical and chemical changes in real time under realistic operating conditions is key to identifying degradation pathways and guiding the design of more durable electrodes and electrolytes.

Imaging techniques have long provided essential insights into structural and chemical heterogeneities in materials. When coupled with spatially and temporally resolved synchrotron-based X-ray analytical tools, such as diffraction, absorption and fluorescence spectroscopies, a direct assessment of the dynamics of a material's activity under working conditions is possible. *Operando* chemical imaging therefore offers a robust approach for probing degradation processes as they unfold, revealing complex interactions that govern electrochemical performance.

Synchrotron X-ray microdiffraction imaging with micrometre resolution over a scanned area of 120 µm × 120 µm, allows the structural and chemical variations to be captured in real time during cycling of an all-solid-state battery[1] (Fig.1).

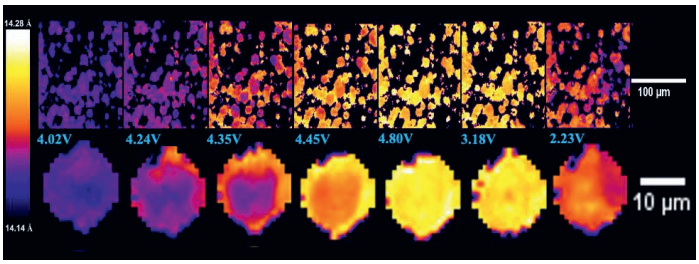


Fig. 1. *Operando* synchrotron-based chemical imaging obtained using µXRD showing the spatial distribution of Li-rich NMC (top), and a zoomed in view of a single spherical particle showing unit cell c lattice parameter (bottom). When lithium is extracted or inserted, the particle contracts or expands accordingly.

Central to these results was a multipurpose, custom-designed electrochemical cell, optimized for X-ray chemical imaging (Fig. 2). It enabled testing of battery materials under tightly regulated environmental conditions (*e.g.* inert gas or controlled humidity), improving reproducibility and protecting sensitive components during the experiments. With this setup, we uncovered heterogeneous lithiation and delithiation behaviours among tens of individual Li-rich NMC (Nickel, Manganese, Cobalt, Oxide) ($Li_y(Ni_{0.19}Mn_{0.54}Co_{0.1})O_{2-x}$) particles. The observations revealed evidence of varied lithium diffusion pathways, reversible formation of intermediate phases, and parasitic reactions linked to trace water contamination.

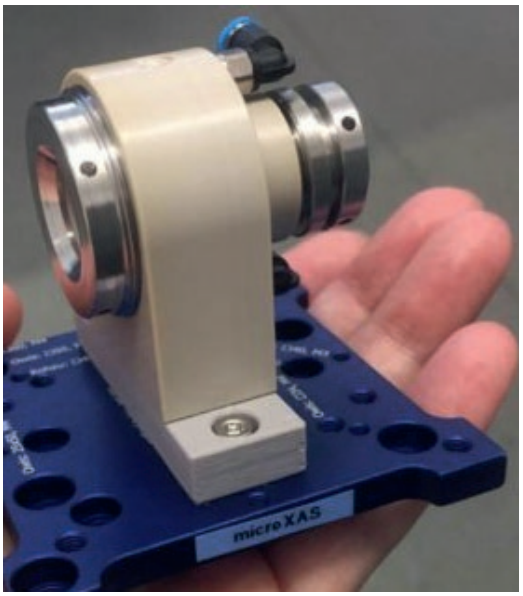


Fig. 2. Controlled-atmosphere battery test cell designed at microXAS for precise electrochemical measurements.

Beyond Li-ion systems, this *operando* imaging methodology can be extended to other electrochemical technologies including Na-ion, Zn- and Li–air,[2] and Li–S batteries. **By bridging the analysis of solid, liquid, and gas phase processes, *operando* X-ray chemical imaging paves the way for a deeper understanding of degradation phenomena and contributes to the development of more reliable next-generation energy storage systems.**

Received: April 24, 2026

1. C. G. Anchieta, B. Lelotte, H. V. Ramasamy, A. Forster, A. Coskun, M. El Kazzi, D. F. Sanchez, *Small Methods* **2025**, *10*, e01793, https://doi.org/10.1002/smtd.202501793.
2. C. G. Anchieta, B. A. B. Francisco, J. P. O. Júlio, P. Trtik, A. Bonnin, G. Doubek, D. F. Sanchez, *Small Methods* **2024**, *8*, 2301749, https://doi.org/10.1002/smtd.202301749.

Can you show us your analytical highlight?
Please contact: Dr. Bodo Hattendorf, ETH Zurich, HCI G105, Vladimir-Prelog-Weg 1, CH-8093 Zurich, Tel.: +41 44 632 44 72
E-mail: analytical_highlights@chimia.ch