Dear participants,

Unfortunately, the 2020 EPDIC conference had to be postponed to 2021 due to the unpredictable and unfortunate situation with COVID-19. We regret that we will not be able to discuss face-to-face and exchange ideas on the latest achievements in science and new technology trends.

Nevertheless, we are pleased to **Welcome you to Bruker's digital luncheon!**

Look forward to our originally planned contributions, covering topics from line-profile analysis, to PDF analysis and thin film characterization:

- In-operando Characterization of $LiNi_xMn_yCo_zO_2$ Pouch Cells
- Structural characterization of ultra-thin metallic films using coplanar and non-coplanar grazing incidence diffraction geometries
- Joint PDF - Rietveld refinements of $LiMn_2O_4$ from laboratory data
- Application of Machine Learning to XRD Phase Identification
- An efficient Rietveld compatible approach to (an)isotropic micro-strain broadening introduced by linearly correlated metric distributions

Use the opportunity to get in touch with our experts after the presentation during the **Q&A session** and stay tuned to hear:

What's new at Bruker?

Be the first to know! We will share with you our latest innovations in X-ray diffraction technology – from single components to entire diffraction solutions. Find out which recent innovations will make a difference to your work and how Bruker is supporting you throughout and beyond the COVID-19 pandemic.

We look forward to seeing you!

Kind regards,
Your Bruker XRD Team

In-operando Characterization of LiNixMnyCozO2 Pouch Cells

Christina Drathen

Since their commercial introduction beginning of the '90ties, small-size lithium-ion batteries have found wide-spread use in consumer electronics due to their high specific energy and energy density. Recently lithium-ion batteries have also started replacing nickel-metal hydride batteries in hybrid and electric vehicles. These automotive applications require large-size batteries with high energy density and superb cycle life time. The cathode material has a major impact on the battery capacity. Therefore, improving batteries for automotive and power grid applications is very much focused on researching cathode materials and their characteristics, especially under in-operando conditions. In this context, pouch cells have become an industry standard battery design due to their efficient shape and lightweight construction.

Here we present the in-operando characterization of a $LiNi_xMn_yCo_zO_2$ (NMC) pouch cells on a laboratory diffractometer equipped with Mo-radiation and a large 2D detector. A transmission geometry XRD set-up was used that allows simultaneous monitoring of both cathode and anode of the pouch cell for cycling effects. Diffraction data were collected continuously whilst cycling at C/5 rate, resulting in 400 diffraction patterns over two charge/ discharge cycles. The diffraction data were analysed using DIFFRAC.TOPAS in batch mode, taking the sandwiched layer cell design into account to include peak intensity, profile and position corrections. The fine time slicing provides detailed insight on the structural changes happening during the cycling process on both anode- as well as cathode.

RiseSun MGL is acknowledged for the kind provision of the NMC pouch cell.

Structural characterization of ultra-thin metal films using coplanar and non-coplanar grazing incidence diffraction geometries

Hugues Guerault

Symmetric diffraction geometries remain reasonable approaches for the analysis of coatings (micrometre range) and thick films (sub-micrometre range). Intensity limits are however reached when the diffraction volume gets too small (<100 nm) with an X-ray penetration depth significantly larger than the layer thickness. For thin films, and down to ultra-thin films (<10 nm), the accessible diffraction volume is very limited and grazing incidence diffraction techniques enable the enhancement of the signal through the control of the penetration depth. The confinement into (ultra-)thin film leads to highly anisotropic properties and emphasizes parameters like preferred orientation, residual stress, micro-strains or crystallite size and shape. Coplanar and non-coplanar grazing incidence geometries probe the film properties in different directions and provide complementary information for an overall understanding of the film structure.

Ultra-thin metal films have been investigated using a newly released BRUKER AXS diffractometer (D8 DISCOVER with non-coplanar arm). Diffraction patterns were collected up to very high angular range to ensure best accuracy on lattice spacing and microstructural parameters. A combined refinement of all dataset based on Whole Powder Pattern Fitting using TOPAS software leads to a consistent evaluation of stress-free lattice parameter, residual stress, micro-strains and crystallite size/shape.

Joint PDF - Rietveld refinements of $LiMn_2O_4$ from laboratory data

Michael Evans

Joint global Rietveld refinements to take advantage of complimentary information available in X-ray and neutron data is a well-established methodology in structure determination and refinement. While analysis of Bragg data can only provide information of the average structure, joint PDF - global Rietveld refinements add the ability to simultaneously refine average and local structure models taking advantage of complementary PDF and (X-ray or neutron) Bragg diffraction data from powders or single crystals.

Here we present the characterization of $LiMn_2O_4$ through combined PDF and Rietveld refinements from data collected on a laboratory diffractometer equipped with a large 2D detector. $LiMn_2O_4$ is a well-known cathode material for Li-ion batteries, whose average structure can be described as a cubic spinel with 6 equivalent Mn-O bonds. However, a Jahn-Teller type distortion influences the local structure that is not easily observable in Rietveld refinement, but which can be seen in the PDF. Previous studies examining the local structure were done using neutrons [1] or with synchrotron X-rays [2]. Data suitable for PDF and Rietveld refinements could be obtained on a laboratory instrument in measurement times as short as 1 hour, and the flexibility of detector positioning allows for optimizing the measurement for maximum resolution or intensity. The raw data and the PDF were analysed using DIFFRAC.TOPAS.

[1] Kodama, K. et al., J. Phys. Soc. Jpn. 82, 094601 (2013)

[2] Yavuz, M. (2016), Investigation of Local and Average Structure in Li-ion Battery Electrode Materials by X-ray Diffraction, PhD Thesis

Application of Deep Learning to XRD Phase Identification

Jan Schützke

Identification of crystalline phases in mixtures is a frequently performed task in powder XRD. It usually involves some software for searching databases of known compounds and matching lists of d-spacings and related intensities to the measured (or reduced) data. Figures-of-merit are usually taken as numerical indicators for the probability of the individual phase assignments. However, some expertise of the skilled user is still required for a “manual” validation of the results. This is time-consuming and error prone. Automated search/match procedures that mostly apply some iterative procedure of the above aim at making the validation step redundant, but failed proofing to be generally reliable yet.

In recent years, deep learning models established their status as a state-of-the-art approach for automated image analysis, such as detecting cars and pedestrians in a street scene.In analogy, neural networks were applied here for automated phase identification from one-dimensional XRD data. Since measured and labeled scans are only scarcely available and deep learning approaches require an extensive dataset, a framework for the efficient generation of hundreds of thousands of synthetic scans has been developed. For the simulation we used phases and mixtures from the Bruker AXS iron ore and cement application packages that are in wide commercial use.

The trained networks achieve an accuracy of close to 100% for synthetic mixtures of both application packages while analyzing hundreds of scans in under a second, thus outperforming the experts in speed without sacrificing an accurate prediction. Additionally, the models have been tested on measured XRD-scans to confirm the results.

An efficient Rietveld compatible approach to (an)isotropic microstrain broadening introduced by linearly correlated metric distributions

Dominque Ectors

Anisotropic microstrain broadening can occur as a result of many different defect types, inhomogeneities of the crystalline lattice or even experimental conditions. Frequently the interpretation of such effects is greatly complicated by the use of phenomenological models to account for the resulting anisotropic peak broadening. Interpretation of the underlying effect from “fit”-parameters and angular dependencies of the microstrain broadening as shown in Figure 1 is always not trivial.

A comprehensive approach starting from the idea of an approximately linearly correlated (not necessarily symmetric) distribution of individual (real or reciprocal) lattice parameters is proposed. Important benefits in terms of direct interpretability of refinement parameters, the ability to constrain parameters to reasonable value-ranges, parameter efficiency as well as direct identification of isotropic microstrain distribution from the fit parameters are the results of this approach. The practical implementation into the TOPAS Rietveld framework is discussed and validated against experimental data.