

MS40-1-8 Tracking the high-temperature synthesis of LiNiO₂ under oxygen gas flow by laboratory-based X-ray diffraction

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D. Weber¹, H. Gesswein², M. Bianchini³, T. Brezesinski¹

¹Karlsruhe Institute of Technology, Institute for Applied Materials - Eggenstein-Leopoldshafen (Germany),

²Karlsruhe Institute of Technology, Institute for Applied Materials - Eggenstein-Leopoldshafen (Germany) - Eggenstein-Leopoldshafen (Germany), ³University of Bayreuth - Bayreuth (Germany)

Abstract

Tracking the formation process of cathode active materials for lithium ion batteries can shed a light on product properties and the role of dopants during crystallization in this highly relevant class of materials. The mechanism of formation can be elucidated directly via high temperature in situ synchrotron experimentation under gas flow or by indirect inference over a series of synthesis. Both methods are costly, as synchrotron time is precious and a large number of solid state synthesis can easily occupy experimenters for weeks.

Here, we describe a gas flow reactor for in situ diffraction (GRID) on a laboratory X-ray diffractometer based on a modified design by Chupas *et al.*^[1] including initial studies on the synthesis of LiNiO₂.^[2] As a product, LiNiO₂ that is highly sensitive to small variations in Li⁺ content as well as oxygen partial pressure during synthesis.^[3] Utilizing an in situ reactor on a laboratory diffractometer allows iterating experimental conditions, which is critical in order to observe the formation of good materials. Thus, the loss of resolution compared to synchrotron experiments can be offset with improved measurement procedures specifically adjusted to the material at hand. Standardized measurement procedures and cross instrument calibration allow knowledge transfer from the in situ experiment directly to lab-based bulk synthesis. Observables include the lattice parameters as well as crystallite size broadening at the initial stages of crystallization up to approximately 50 nm. Additionally, we performed a series of isothermal kinetic experiments, thus quantifying the energetic barrier for crystal growth in two regimes. Our study shows opportunities and challenges in lab-based high temperature in situ diffraction measurements and highlights possible pathways towards higher resolution in time and space in PXRD at the lab scale.

References

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Elucidation of high T reaction mechanisms.

