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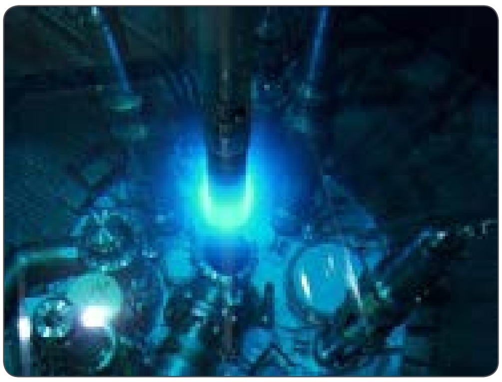
How neutrons as a probe for *in situ* and *in operando* measurements support the understanding of electrochemistry in Li-ion battery research

For a better understanding of the electrochemistry in batteries a huge demand emerge for *in situ* and *in operando* characterization methods. Due to the high penetration depth and high sensitivity of neutrons to light elements as lithium such a probe is more and more attractive in the last decade. This contribution gives an overview how neutrons with their unique properties contribute in the development of new battery cells. During charging and discharging of NMC/graphite cells the intercalation of Li in the graphite layers can be observed *in situ* with neutron diffraction (ND) as such measurements are sensitive to detect LiCx phases as LiC_6 and $LiC1_2$ during the intercalation/de-intercalation process. Under fast charging conditions and low temperatures the appearance of Li plating can be studied. A correlation of C-rates and Li plating is investigated by means of voltage relaxation and *in situ* ND. Batteries consisting of lithium iron phosphate (LFP) are often used for stationary energy storage systems. Here neutrons provide the answer why various types of graphite result in losses of the storage capacity. On larger scales of >50 micrometer neutron imaging (radiography and tomography) enables a non-destructive view inside the cell to make visible for example how the electrolyte filling with the distribution of the electrolyte in the cell between the layer stacks in a pouch cell takes place. The use of neutron induced prompt gamma activation analysis (PGAA) is a powerful tool to describe the capacity loss of the cell caused by tiny metal deposition on the graphite anode after charging/discharging processes. The method of neutron depth profiling (NDP) is suited to study near surface phenomena as the Li distribution in electrodes. A new set-up for NDP is currently under development to improve the space resolution and to measure with a time resolved mode.



Recent Publications

1. Zinth V, V Lüders C, Hofmann M, Hattendorf J, Buchberger I, Erhard S V, Rebelo Kornmeier J, Jossen A and Gilles R (2014) Lithium plating in lithium-ion batteries at sub-ambient temperatures investigated by *in situ* neutron diffraction. Journal of Power Sources 271:152-159.
2. V Lüders C, Zinth V, Erhard S V, Osswald P J, Hofmann M, Gilles R and Jossen A (2017) Lithium plating in lithium-ion batteries investigated by voltage relaxation and *in situ* neutron diffraction. Journal of Power Sources 342:17-23.

3. Paul N, Wandt J, Seidlmayer S, Schebesta S, Mühlbauer M J, Dolotko O, Gasteiger H A and Gilles R (2017) Aging behavior in $LiFePO_4$/C 18650-type cells studied by *in situ* neutron diffraction. Journal of Power Sources 345:85-96.
4. Knoche T, Zinth V, Schulz M, Schnell J, Gilles R and Reinhart G (2017) *In situ* visualization of the electrolyte solvent filling process by neutron radiography. Journal of Power Sources 331:267-276.
5. Buchberger I, Seidlmayer S, Pokharel A, Piana M, Hattendorff J, Kudejova P, Gilles R and Gasteiger H A (2015) Aging analysis of graphite/LiNi1/3Mn1/3O2 cells using XRD, PGAA, and AC impedance. Journal of the Electrochemical Society 162(14):A2737-2746.

Biography

Ralph Gilles a Senior Scientist has his expertise in neutron scattering methods for studying energy materials as batteries and high-temperature alloys. Especially, the use of *in situ*, *in operando* methods (very often combined with non-destructive measurements) on real bulk samples enables a powerful tool on energy related topics. In his group methods as neutron diffraction, small-angle neutron scattering, grazing incidence small-angle neutron scattering, imaging, neutron depth profiling and neutron induced prompt gamma activation analysis are applied for battery research. He is an Industrial Coordinator of Heinz Maier-Leibnitz Zentrum, Coordinator of the Materials Science group and Head of the Materials Science Laboratory.

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