X-Ray Spectrometry for the analysis and the speciation of nano- and microporous Li-S cathode materials

Burkhard Beckhoff and Matthias Müller

Physikalisch-Technische Bundesanstalt, Röntgen- und IR-Spektrometrie
Abbestraße 2-12, D-10587 Berlin

The further development of porous cathode materials for Li-S batteries requires a better and quantitative understanding of the material properties, which lead to the limiting electrochemical behavior. To probe physical and chemical properties such as elemental composition, layer thickness or chemical binding states we employ non-destructive X-Ray Spectrometry (XRS) ensuring a characterization without any modification of the sample system. This allows for a reliable correlation of the obtained physical and chemical properties with the electrochemical behavior investigated by electrical measurements on the same samples.

We have developed synchrotron radiation based XRS methodologies involving well-known radiation sources and radiometrically calibrated instrumentation (photodiodes and energy-dispersive fluorescence detectors) allowing for reference-free quantification schemes [1]. The reference-free scheme enables a reliable quantification of mass depositions, (buried) nanolayer thicknesses as well as elemental and species depth-profiles without relying on calibration standards or reference materials, which are rarely available for novel materials [2].

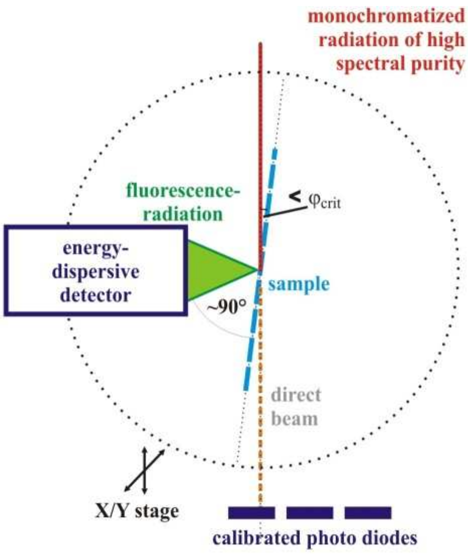

Figure: Experimental set-up in UHV chamber for reference-free XRS in grazing incidence geometry.

Employing reference-free XRS, we investigate the mass deposition of sulfur intercalated in the porous cathode material and our partners are using these results to optimize the deposition process of nano-scaled sulfur objects into block co-polymers. Furthermore, we aim at the quantitative determination of sulfur depth profiles by means of angular dependent XRS measurements in grazing incidence geometry in order to further increase the understanding of the sulfur intercalation process. In addition to reference-free XRS, we use x-ray absorption spectrometry [3] to investigate the binding state of intercalated sulfur. The chemical speciation of sulfur aims at the identification of unintentional sulfur species, which may have a negative impact on the charge and discharge reversibility of Li-S batteries.

[1] B. Beckhoff, R. Fliegauf, M. Kolbe, M. Müller, J. Weser, G. Ulm, Anal. Chem. (2007) **79**, 7873.

[2] R. Unterumsberger, B. Pollakowski, M. Müller, B. Beckhoff, Anal. Chem. (2011) **83**, 8623.

[3] C. Fleischmann, S. Sioncke, S. Couet, K. Schouteden, B. Beckhoff, M. Müller, P. Hönicke, M. Kolbe, C. Van Haesendonck, M. Meuris, K. Temst, A. Vantomme, J. Electrochem. Soc. (2011) **158**, H589.