X-RAY FLUORESCENCE ELEMENTAL IMAGING, MICRO ANALYSIS AND
SPECIATION OF AGED LI-ION CELL ELECTRODES

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Micro X-ray fluorescence (MXRF) allows for spatial resolved elemental analysis and imaging of condensed matter. X-ray probes in general provide high penetration depth and therefore allow for 3D observation of representative parts of a system e.g. battery electrodes. Modern synchrotron sources easily offer spatial resolutions of ca. 100-500 nm. Whereas laboratory based instruments typically have a spatial resolution in the mesoscopic range i.e. 10-30 µm focal diameter size. Using micro-X-ray fluorescence and micro-XANES at a synchrotron source, changes in elemental distribution and redox species correlated to the aging process of LiNi0.5Mn1.5O4 Li-ion battery electrodes were studied with a spatial resolution of 0.5 µm at PetraIII P06 (DESY, Hamburg, Germany) [1]. For comparison the same electrodes were also imaged using scanning electron microscopy X-ray emission analysis (SEM/WDS). The method confirmed the presence of Ni “hot spots” in the aged electrode. However, the SEM/WDS measurements were hampered by the roughness of the composite electrode and the measuring time had to be extended to account for the low count rates of Ni and Mn Kα lines.

Lab based 3D confocal micro-XRF (CMXRF) laboratory based instrumentation will be established at WSU in the near future and will allow for spatial resolution of ca. 20x20x20µm3.

[1] U. Boesenberg, M. Falk, C. G. Ryan, R. Kirkham, M. Menzel, J. Janek, M. Fröba, G. Falkenberg, U. Fittschen, Correlation between chemical and morphological heterogeneities in LiNi0.5Mn1.5O4 spinel composite electrodes (LNMO) for lithium ion batteries determined by micro X-ray fluorescence analysis, Chem. Mater., Just Accepted Manuscript • DOI: 10.1021/acs.chemmater.5b00119