XES Nanoprobe for Hard X-Ray Region

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Pacific Northwest NATIONAL LABORATORY
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Purpose

Build high-spatial-resolution X-ray emission spectroscopy (XES) for in situ analysis of X-ray emission lines in the 5- to 10-keV range. Development of a low-cost, modular XES system with combined nanometer spatial resolution and in situ capability will be unique and widely applicable to fundamental materials chemistry challenges

Key Successes

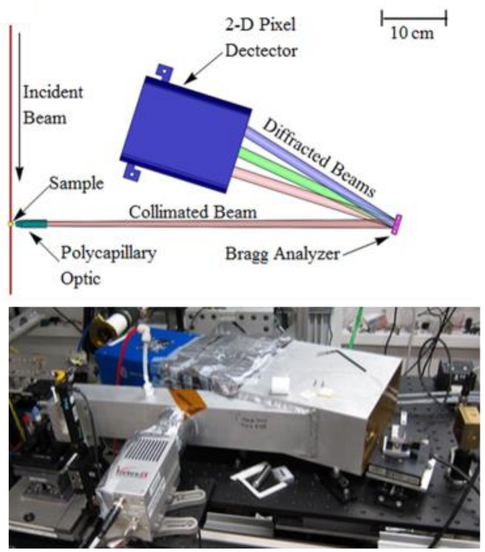
Demonstration of new Ni XES, miniXS, & polycapillary spectrometers at APS 20-ID

- 2D XES and X-ray absorption near-edge structure (XANES) maps of ZEBRA* battery cathode reveal that FeS additive is electrochemically active, and higher FeS additive concentration promotes formation of larger $NiCl_2$ grains

*Zeolite Battery Research Africa Project

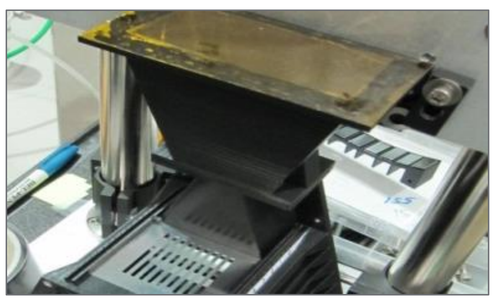
Research Accomplishments

New polycapillary spectrometer for in situ XES



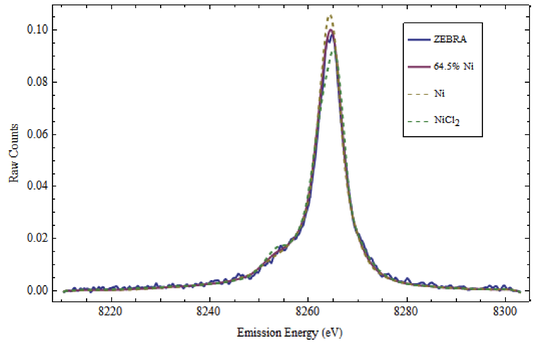
Mortensen et al. 2013. *Review of Scientific Instruments*, submitted.

New Ni miniXS spectrometer for XES

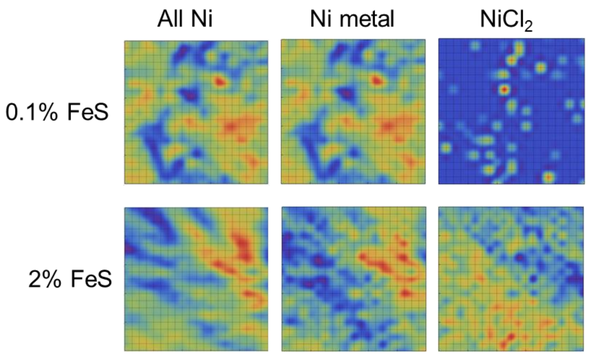


XES mapping of Ni-ZEBRA battery cathode

- XES provides elemental abundance and more detailed chemical speciation (ligands, spin and oxidation states) compared to XAS
  - Maps collected in a single raster scan, eliminating registration problems with motor backlash
- Early results suggest size of $NiCl_2$ particles increases with increasing FeS concentration



Relative Ni and $NiCl_2$ derived by linear combination fitting of XES spectra

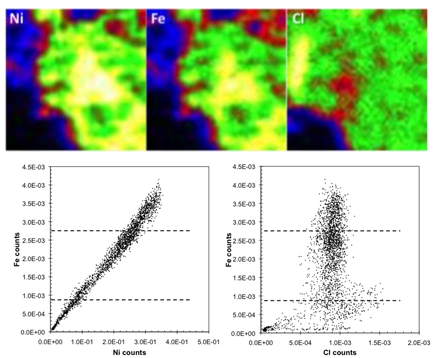


100 µm X 100 µm XES maps

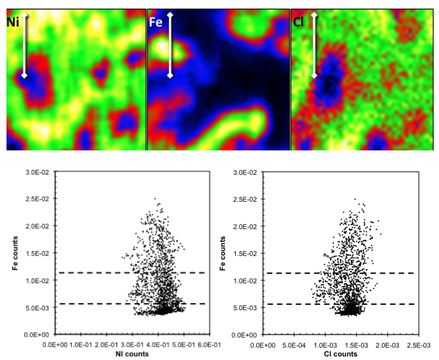
XAS mapping of Ni-ZEBRA battery cathode

- X-ray fluorescence mapping provides elemental abundance
- Results indicate Fe-Ni spatial correlation only occurs in the charged state

charged state



discharged state



Bowden et al. 2013. *Journal of Power Sources*, submitted.

Next Steps

- Detailed understanding of how Fe prolongs ZEBRA battery life
- In situ XES study of lithiation of lithium manganese nickel oxide (LMNO) battery system with CII Project 1.9
- Spectroscopic tomography of ZEBRA cathode at SSRL
- Leverage DOE-BES investments in the Joint Center for Energy Storage Research