TENDER-ENERGY SPATIALLY RESOLVED AND *IN-SITU* X-RAY ABSORPTION SPECTROSCOPY (TES)

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II

SCIENTIFIC SCOPE

TES is a beamline for high performance and *in-situ* X-ray absorption spectroscopy and spatially-resolved XAS and imaging of heterogeneous or structured materials; it is optimized for the "tender" energy range from 1-5 keV (reaching up to 8 keV) in a helium (non-vacuum) environment. Two endstations are optimized for 1) microbeam EXAFS and X-ray fluorescence imaging and *in-situ* fuel-cell and battery measurements, and 2) chemistry and catalysis *in-situ* and *in-operando* XAFS. Fast on-the-fly scanning of the stage and monochromator enables rapid imaging and XAS of dynamic systems.

Overview

PORT: 8-BM
SOURCE: Dipole Bend Magnet
ENERGY RANGE: 1 – 8 keV
ENERGY RESOLUTION: $\Delta E/E = 10^{-4}$
SPATIAL RESOLUTION: ~1 μm to ~1 mm
FAST ENERGY SCANNING: 1 to 75 eV/s
CONSTRUCTION PROJECT: NxtGen
BEAMLINE STATUS: Construction
AVAILABLE TO USERS: 2016

BEAMLINE CHARACTERISTICS

TECHNIQUES:

- X-ray Absorption Spectroscopy
- Microbeam XAS including EXAFS
- In-situ, in-operando and rapid XAS
- XRF and XAS imaging

ENDSTATION DETAILS:

- Achromatic KB Microbeam EXAFS
- X-ray Fluorescence Microprobe
- *In-situ* and *in-operando* fast XAS
- Catalysis and electrochemistry

TES at NSLS-II:

- Will enable **spatially-resolved** and ***in-situ*** studies of speciation and local structure by XAS, X-ray fluorescence and spectroscopic imaging, in a helium *non-vacuum environment*
- Chemical sensitivity to **key lighter elements** Mg through Ti, and advantageous heavier-element L and M edges such as Zr, Cd, Pd, U.

Beamline Team

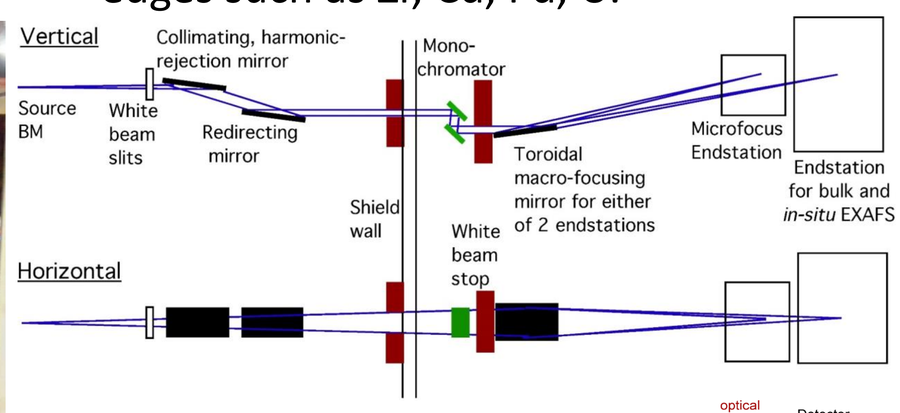
STAFF

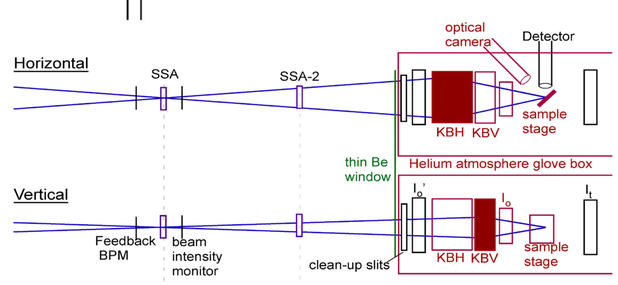
Paul Northrup: lead beamline scientist
Lukas Lienhard: mechanical engineer
Michael Johanson: designer

BEAMLINE DEV. PROPOSAL LEAD

Paul Northrup (Brookhaven National Lab)

Endstation 1, constructed and commissioned at NSLS beamline X15B in 2014. From left, ion chamber, KB mirrors, optical camera, sample stage, Ge and Si tandem detectors, ion chamber.





Versatile Compound Focusing: Layout of TES optics from BM source through endstations.

SCIENTIFIC APPLICATIONS

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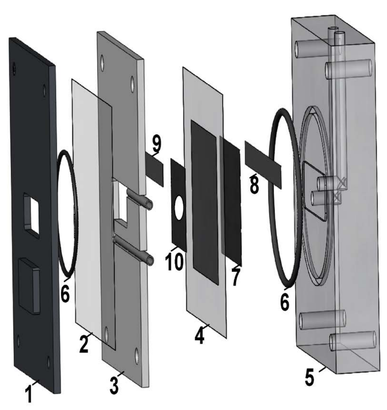
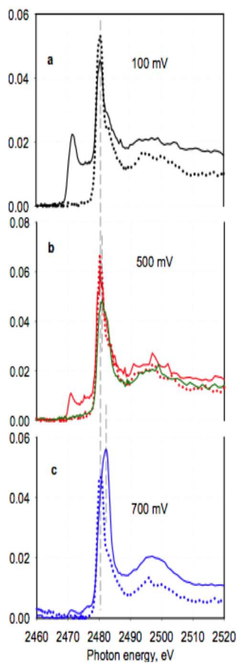
- **Energy Materials**: Photovoltaic, fuel-cell, battery and superconducting (nano)materials.
- **Catalysis**: Materials (zeolites, thin films, nanomaterials), reaction mechanisms and intermediate species, poisoning of catalysts.
- **Environmental/Earth Science**: Biogeochemical and redox processes, contaminant behavior and remediation; Ca-Mg-Si high-pressure phases.
- **Climate**: Terrestrial/marine C cycling, carbonate (bio)mineralization, geologic record of climate change, ocean chemistry, CO_2 sequestration.
- **Sustainability**: Nutrient (P, S, K, Ca, Mg, Fe) cycling, transport and bioavailability, biofuel/biomass productivity, poor or highly-leached tropical soils.

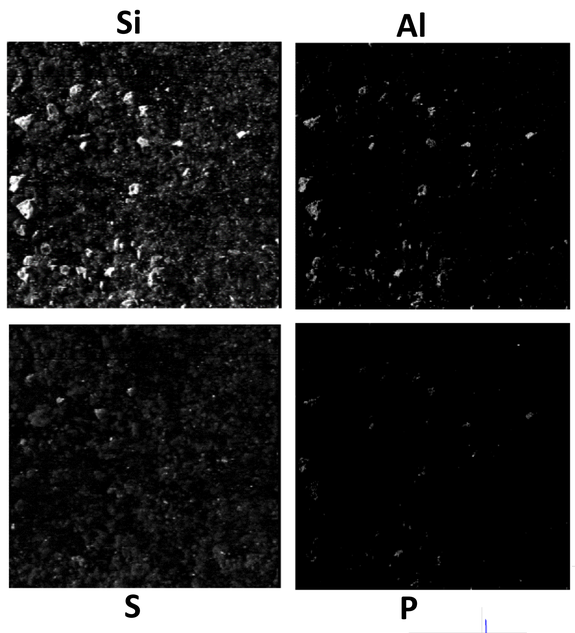
Sulfur poisoning of Fuel-Cell catalysts: *in-situ* XAS

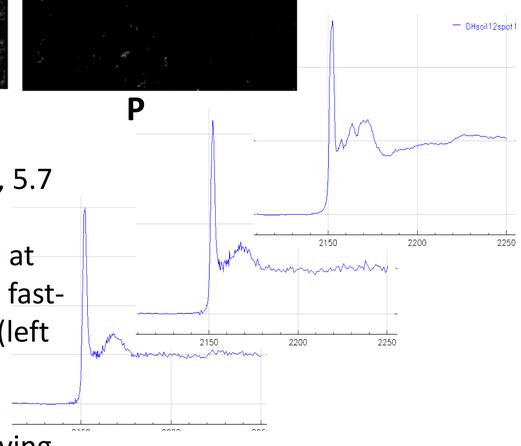
- Novel **Spectroelectrochemical cell** design for *in-situ* measurements of fuel-cell catalyst at controlled electrochemical potential and under gas flow, all compatible with low-energy fluorescence-mode XAS measurements.
- Real fuel-cell materials and geometry.
- Sample spectra under varying potentials, indicating changes in S speciation and oxidation state.





O. Baturina, B. Gould, Y. Garsany, R. Stroman and P. Northrup, "Identification of adsorbed SO_2 species on fuel cell electrocatalysts by sulfur K-edge XANES combined with electrochemistry" in review.





Agricultural soil sample, 5.7 x 5 mm area, highly heterogeneous, imaged at 2800 eV. Microbeam P fast-XANES spectra identify (left to right) adsorbed phosphate on Al/Fe oxyhydroxide, P in decaying organic-matter, and one single grain of Al Phosphate mineral precipitate.

Phosphorus distribution and speciation:

Key nutrient but environmental contaminant: cycling and bioavailability influence global photosynthesis and biofuel/agricultural productivity and sustainability. But behavior depends on local chemical speciation – bulk analyses are not representative.