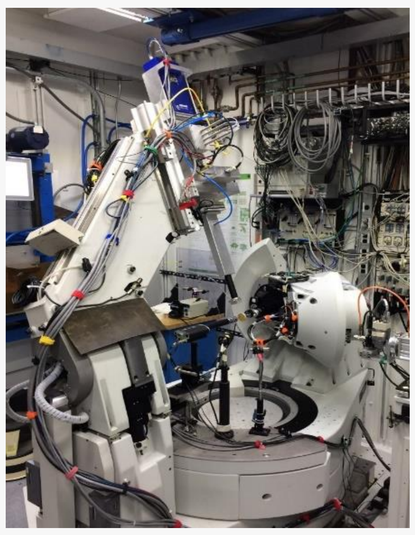
Surface, Interface and Powder Diffraction Program

Joanne E. Stubbs[1] and Peter J. Eng[1,2]

[1]Center for Advanced Radiation Sources, [2]The James Franck Institute -The University of Chicago, Chicago, IL 60637, USA

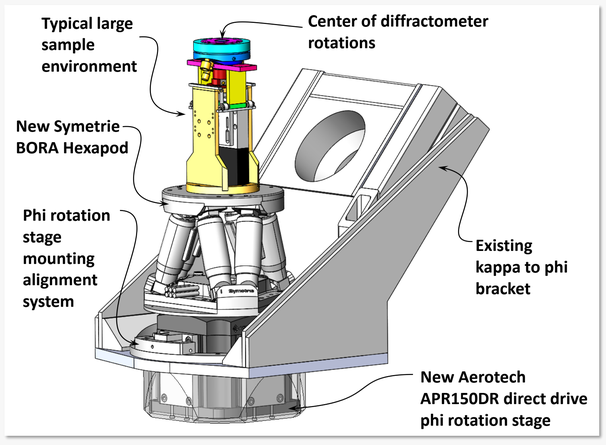
End Station Instrumentation



13-ID-C
- Tunable energy: 5-75 keV
- Focus: < 1 μm after APS-U optics upgrade

13-BM-C
- Fixed energies: 15 & 28.6 keV
- Vertical focus: 30 μm
- Horizontal focus: 300 μm

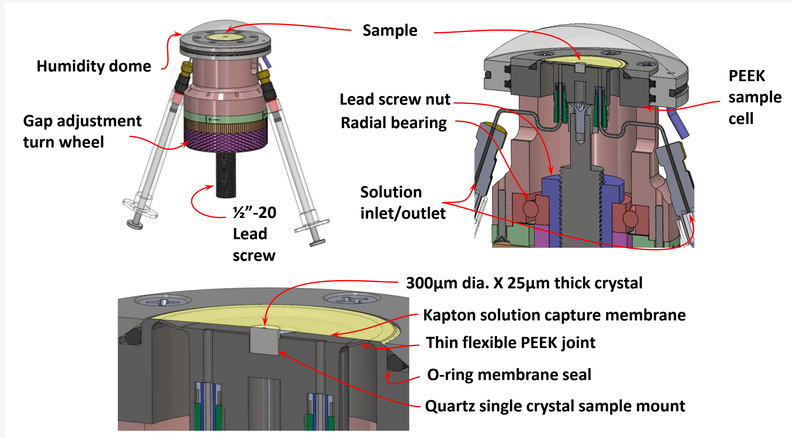
Newport Six-Circle Kappa Diffractometers
- Open cradle – no chi circle
- Large, heavy, complex samples & detectors
- Dectris Pilatus pixel array detectors
- Solid state XRF detectors

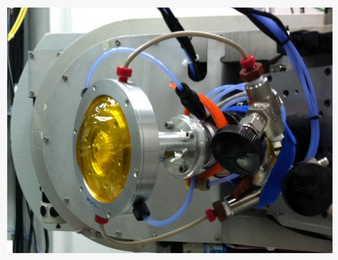


Diffractometer upgrades
- High-speed sample rotation axis (up to 60°/sec)
- Hexapod sample positioning

Custom Specialized Sample Environments

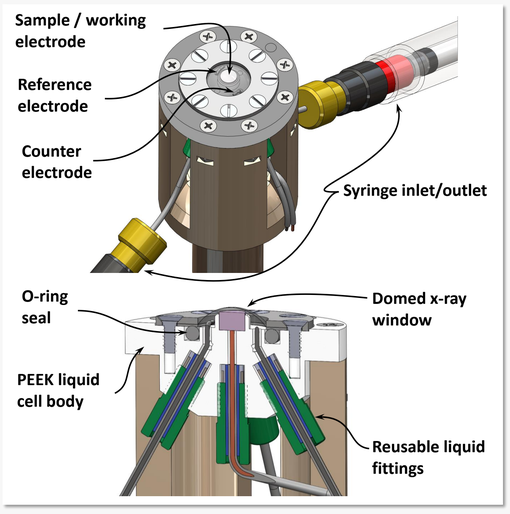
Microcrystal mineral-water interfaces





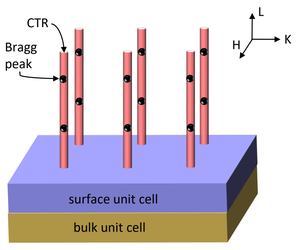
Redox-sensitive gas handling and hazardous sample containment

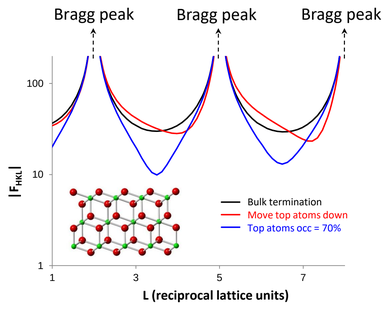
In-situ electrochemistry



Interfacial X-ray Scattering

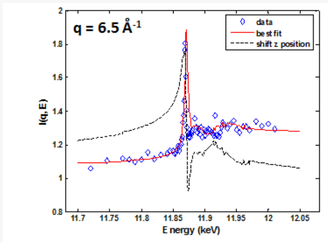
Crystal Truncation Rod (CTR) Diffraction
Atomic-scale surface/interface structures

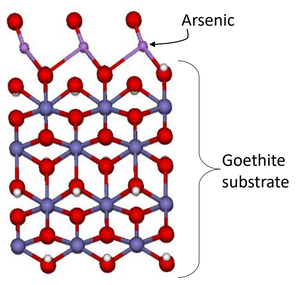




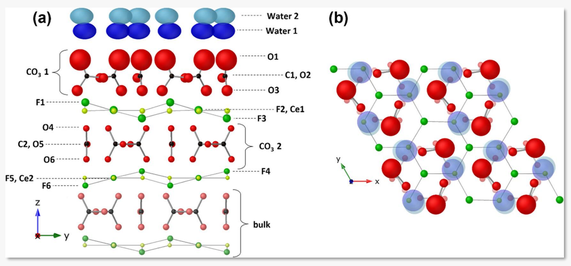
- *Highly sensitive to surface termination*
- *Allows work under liquids and gases*

Resonant Anomalous X-ray Reflectivity (RAXR): Element-specific CTR



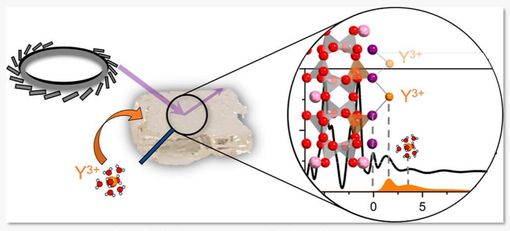


Science Highlight: Structure of the Bastnäsite (001) Surface by Crystal Truncation Rod X-ray Diffracion and Ab Initio Molecular Dynamics: Implications for Separations of a Rare Earth Ore Mineral

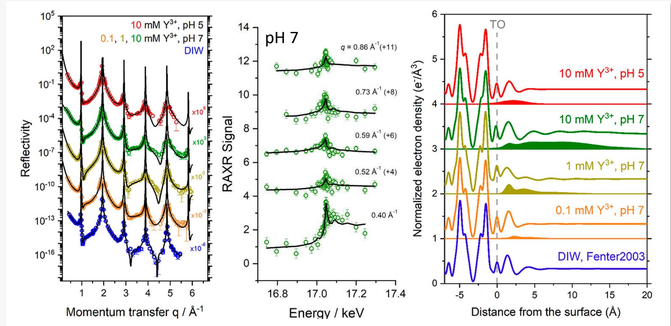


Wanhala et al., 2023, *J. Phys. Chem. C*

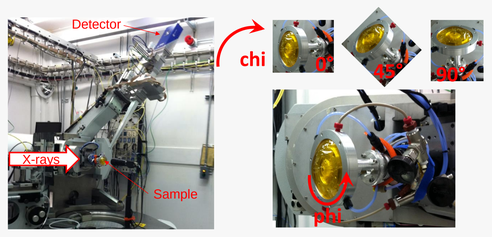
Science Highlight: Y(III) Sorption at the Orthoclase (001) Surface Measured by X-ray Reflectivity



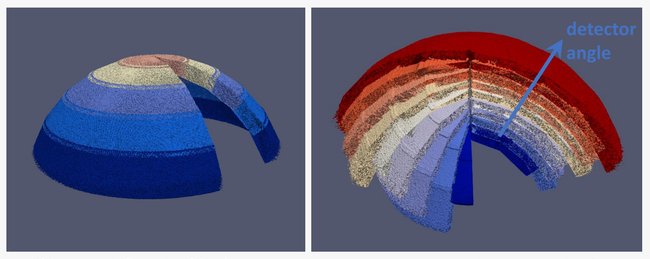
Neumann et al., 2023, *Environ. Sci. Technol.*



Reciprocal Space Maps

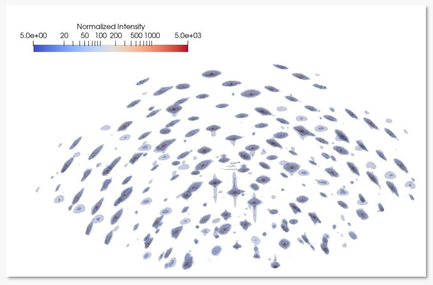


Collect data over range of phi, chi, detector angles

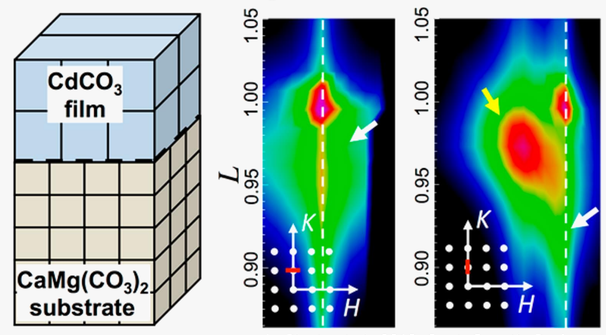


- Phi scans at discrete chi values produce rings
- All scans at given detector angle produce hollow dome
- Width of chi bands, size of phi steps, and thickness of dome governed by detector size
- Repeat at many detector angles for set of concentric, overlapping domes

Reconstruct 3D reciprocal space volume

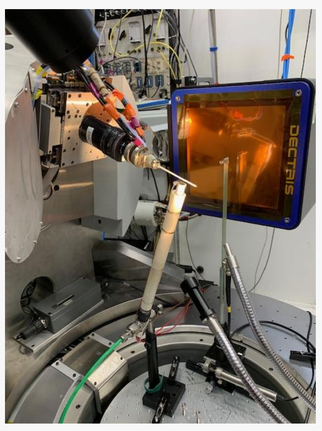


Science Highlight: Evolution of Strain in Heteroepitaxial Cadmium Carbonate Overgrowths on Dolomite



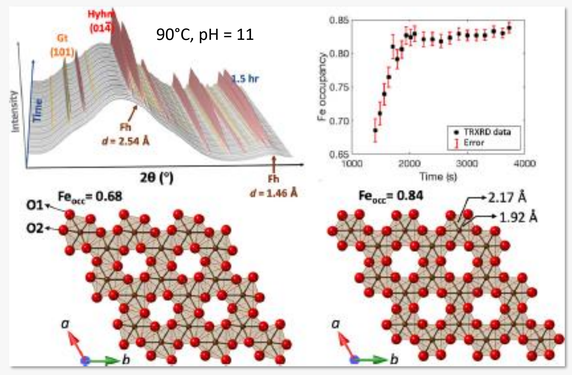
La Plante et al., 2018, *Cryst. Growth. Design*

In-situ powder diffraction



In-situ capillary heating
- Nested ceramic tubes with wire windings for resistive heating
- Flowing He or N_2 gas transfers heat to capillary
- Thermocouple near sample for measurement and feedback
- Temperatures up to 1200°C
- Heating rates up to 200°/sec
- Pilatus3 S 1M detector – up to 25 Hz

Science Highlight: Superhydrous hematite and goethite: A potential water reservoir in the red dust of Mars?

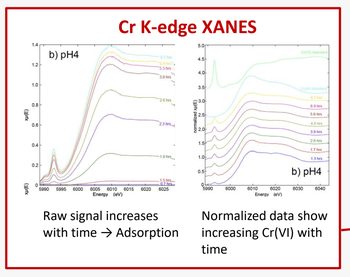


Chen et al., 2021 *Geology*

Flow-through aqueous solution chemistry
- Powdered sample loaded into capillary packed with glass wool
- Aqueous solution flows through capillary
- Reacted solution caught in collection vials for subsequent chemical analysis
- Motorized vial stage advanced automatically, coordinated with collection of diffraction patterns

Synchronous diffraction, XANES, and solution chemistry
- Cr (III) solution flowed through triclinic birnessite powder
- Cr oxidation coincides with triclinic-hexagonal transition
- No transition without Cr
- Both Cr (III) and Cr (VI) adsorb to birnessite

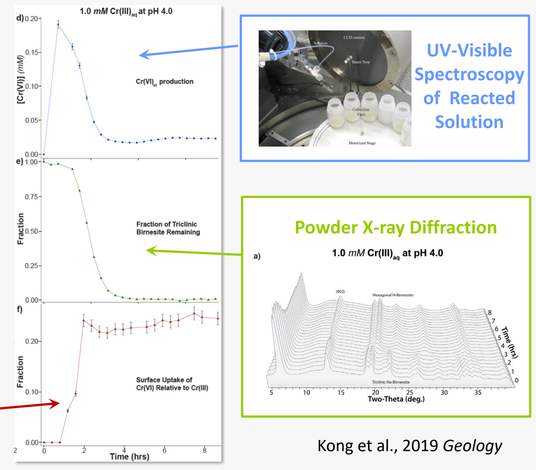
Cr K-edge XANES



Raw signal increases with time → Adsorption

Normalized data show increasing Cr(VI) with time

Science Highlight: Mineralogical and geochemical constraints on chromium oxidation induced by birnessite



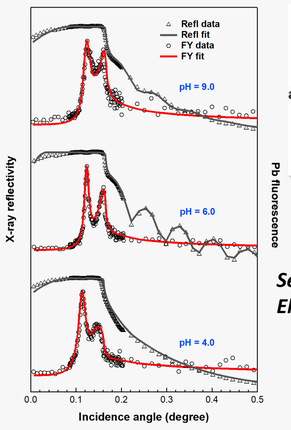
UV-Visible Spectroscopy of Reacted Solution

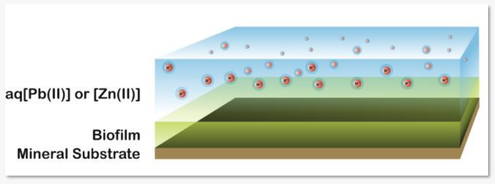
Powder X-ray Diffraction

Kong et al., 2019 *Geology*

Grazing Incidence Spectroscopy

Long Period Standing Wave Fluorescence Yield (LPSW-FY) Spectroscopy

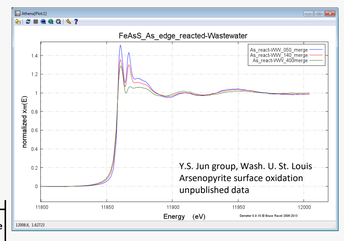




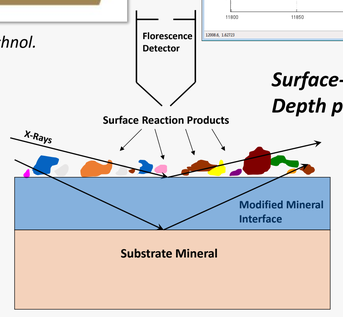
Y. Wang et al., 2013, *Environ. Sci. Technol.*

Sensitive to monolayer coverage
Elemental depth profiling

Grazing Incidence XAFS



Surface-sensitive redox states
Depth profiles



Acknowledgments:

GeoSoilEnviroCARS is supported by the National Science Foundation – Earth Sciences via SEES: Synchrotron Earth and Environmental Science (EAR –2223273). Surface and interface program developments and experiments are further supported by DOE BES Geosciences (DE-SC0019108). This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.