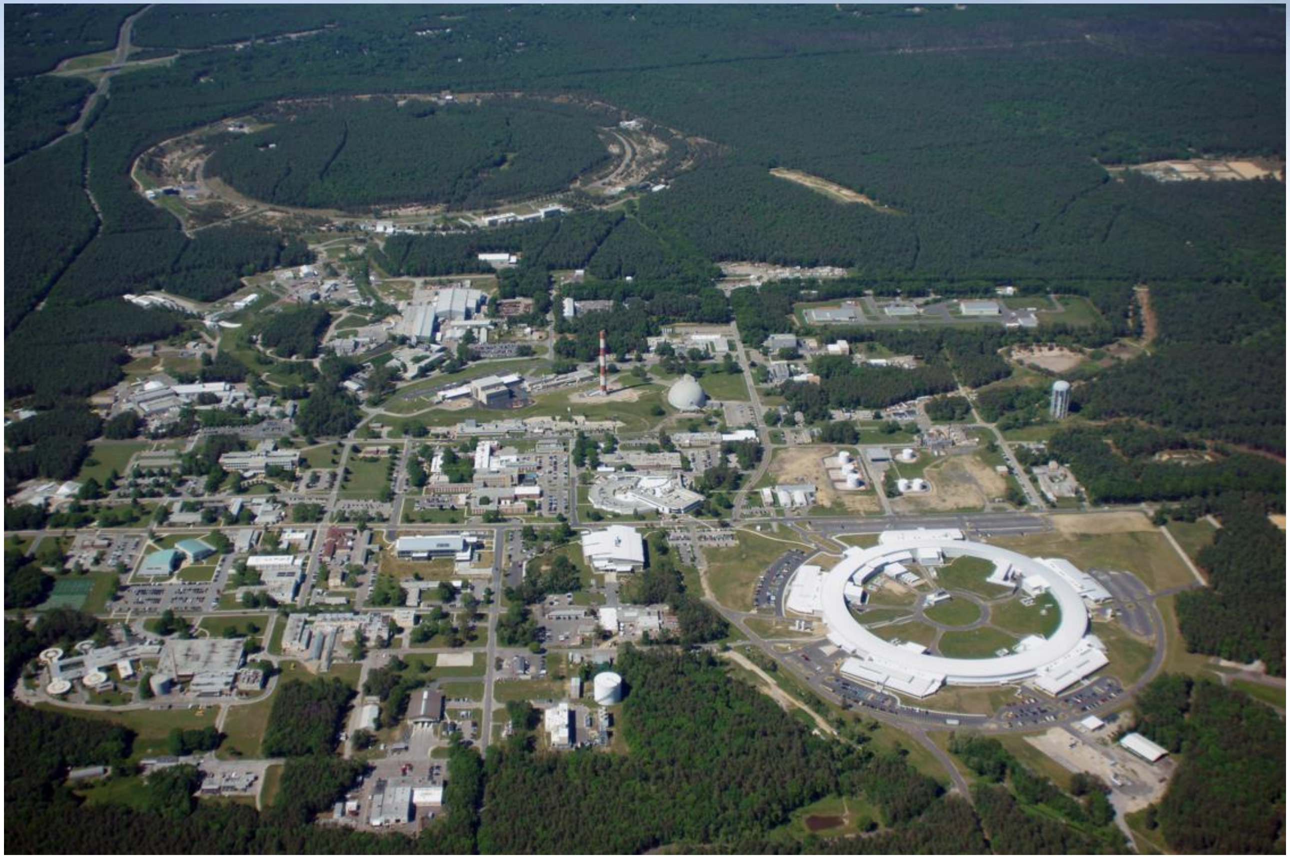


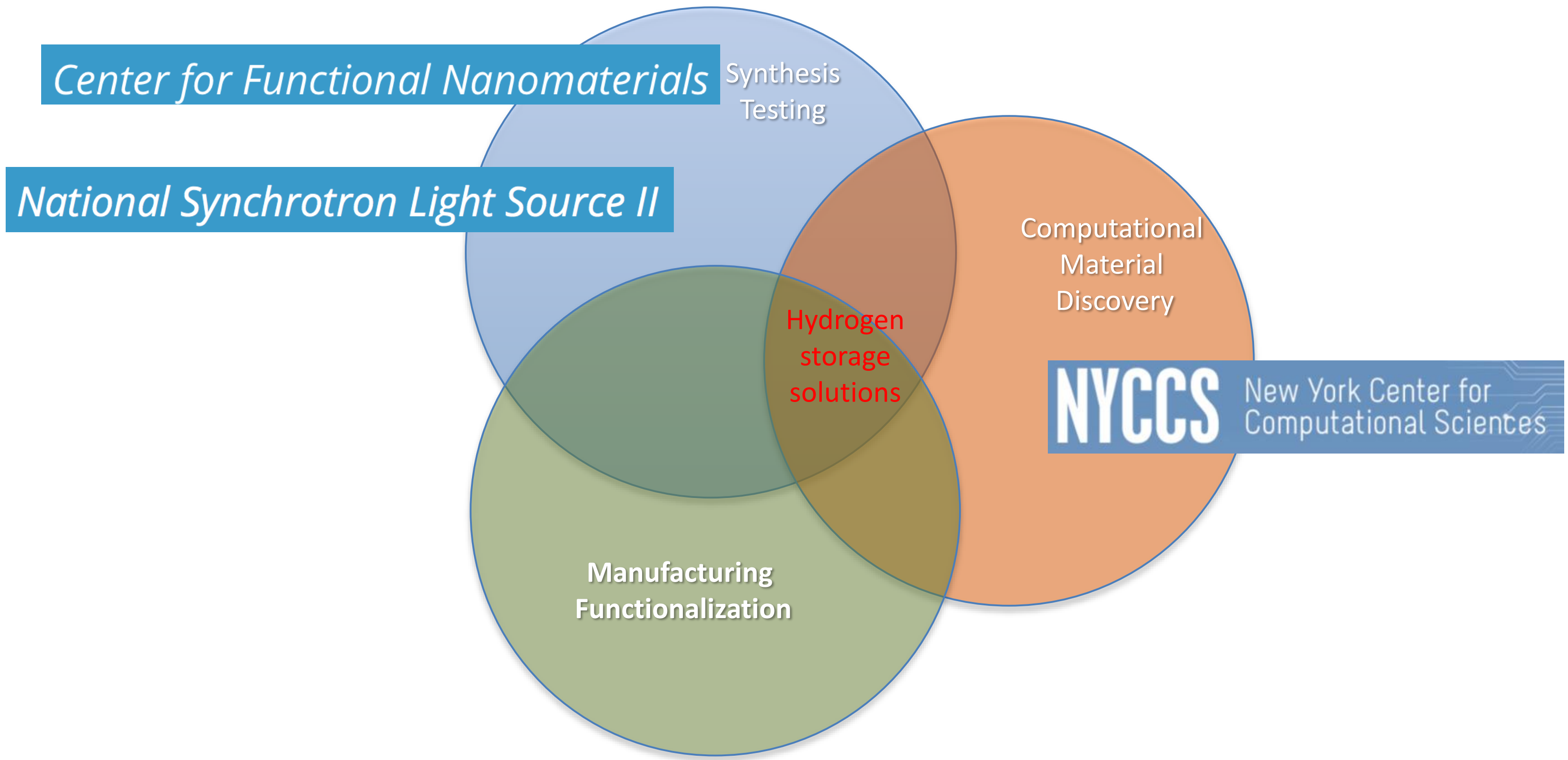
Brookhaven National Laboratory

Eli Stavitski

Laboratory overview

- Brookhaven National Laboratory located on Long Island, NY, is a home to several facilities, relevant to CCS effort
  - National Synchrotron Light Source II is the brightest synchrotron radiation source in the world, in operation since 09/2014, with 6 operating experimental stations and over 20 in constriction.
  - Centre for Functional Nanomaterials: numerous opportunities for material characterization
  - The New York Center for Computational Sciences: access to high performance computing resources

BNL potential to contribute to the mission



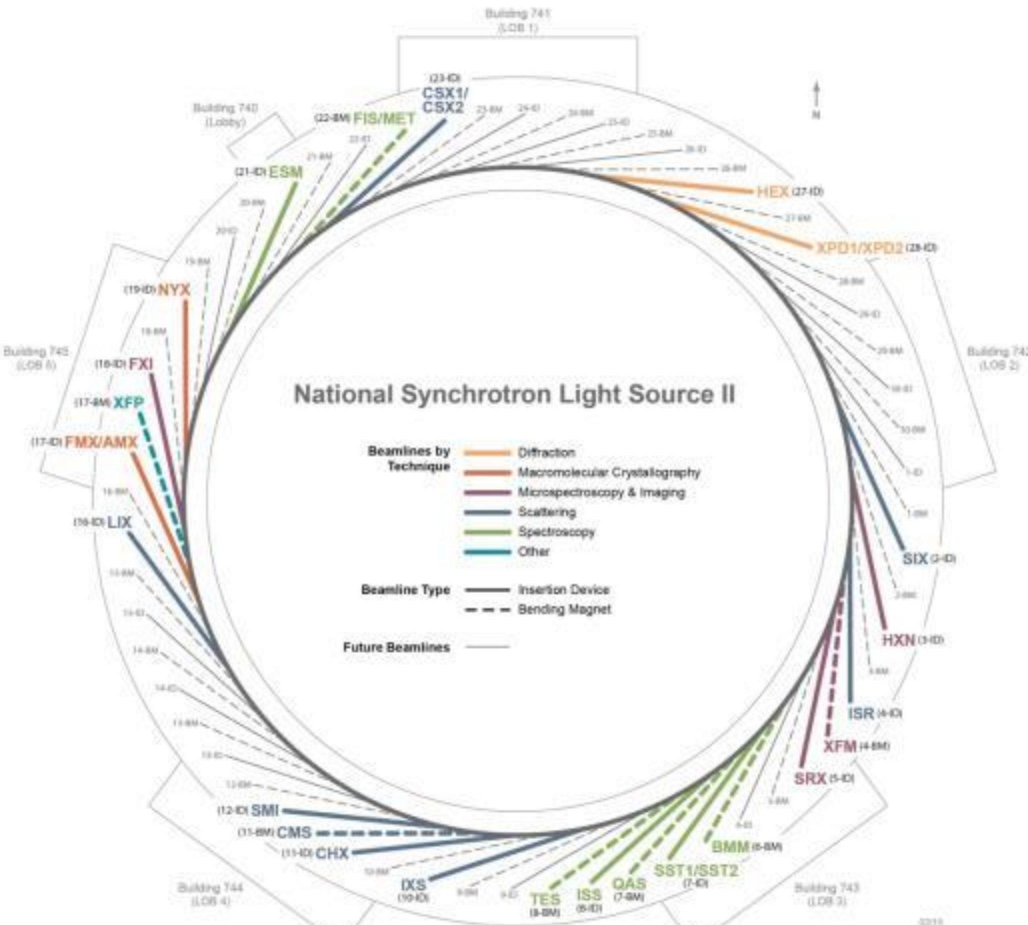
National Synchrotron Light Source-II

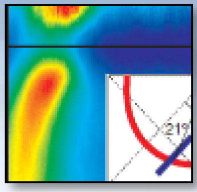
- A third-generation 3GeV light source
- Project (accelerator + 6 beamlines) completed in Sep 2014
- >20 beamlines in construction
- World-leading low-emittance, high brightness, broad spectrum range, large capacity for medium GeV rings, long beamlines, and stability
- Advanced beamlines with cutting-edge optics, detectors, and instrumentation

NSLS-II
Current Suite of Beamlines

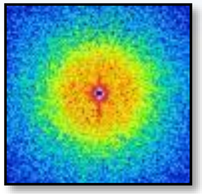
- 6 Operating
- 21 Under Development

http://www.bnl.gov/ps/nsls2/beamlines/map.php

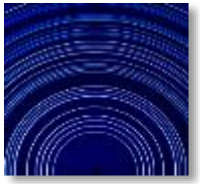




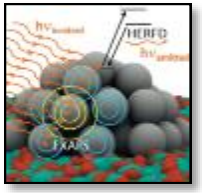
Soft X-Ray Scattering & Spectroscopy
23-ID-1: Coherent Soft X-ray Scattering (2015)
23-ID-2: Soft X-ray Spectr & Polarization (2015)
21-ID: Photoemission-Microscopy Facility (2017)
2-ID: Soft Inelastic X-ray Scattering (2017)
22-BM: Magneto, Ellips, High-P Infrared (2018)



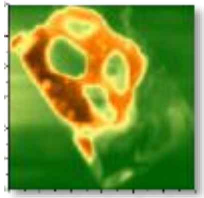
Complex Scattering
10-ID: Inelastic X-ray Scattering (2015)
11-ID: Coherent Hard X-ray Scattering (2015)
11-BM: Complex Materials Scattering (2016)
12-ID: Soft Matter Interfaces (2017)



Diffraction & In Situ Scattering
28-ID-1: X-ray Powder Diffraction (2015)
28-ID-2: X-ray Powder Diffraction (2017)
4-ID: In-Situ & Resonant X-Ray Studies (2017)
27-ID: High Energy X-ray Diffraction (2020)



Hard X-Ray Spectroscopy
8-ID: Inner Shell Spectroscopy (2017)
7-BM: Quick X-ray Absorption and Scat (2016)
8-BM: Tender X-ray Absorption Spectr (2017)
7-ID-1: Spectroscopy Soft and Tender (2017)
7-ID-2: Spectroscopy Soft and Tender (2017)
6-BM: Beamline for Mater. Measurement (2017)

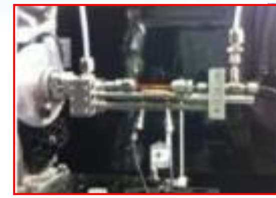


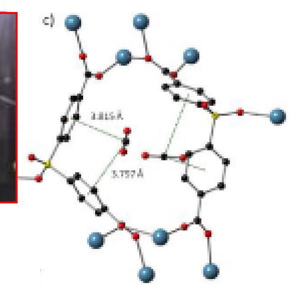
Imaging & Microscopy
3-ID: Hard X-ray Nanoprobe (2015)
5-ID: Sub-micron Resolution X-ray Spectr (2015)
4-BM: X-ray Fluorescence Microscopy (2017)
18-ID: Full-Field X-ray Imaging (2018)

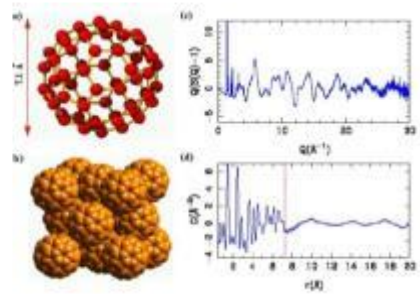
Structural Biology
17-ID-1: Frontier Macromolec Cryst (2016)
17-ID-2: Flexible Access MacromolCryst (2016)
16-ID: X-ray Scattering for Biology (2016)
17-BM: X-ray Footprinting (2016)
19-ID: Microdiffraction Beamline (2017)

In-situ & In-operando Diffraction & Spectroscopy

- X-ray Powder Diffraction (XPD-1/-2) – X-ray powder diffraction beamlines at NSLS-II offer cutting-edge capabilities with a wide suite of in-situ sample cells and real-world in-operando sample environments
- In-situ Resonant Scattering (ISR) – In-situ capabilities include real-time diffraction and scattering in thin-film growth chamber, a 6-350K sample environment, and a gas handling / exhaust system
- Inner Shell Spectroscopy (ISS), Quick Absorption & Scattering (QAS), Tender Energy Spectroscopy (TES), Beamline for Materials Measurements (BMM) – offer *in-situ* specimen environments with hazardous gas handling system for catalysis
- Frontier IR Spectroscopy and Magneto-Ellipsometric & Time-resolved IR Spectroscopy (FIS/MET) enables in-situ optical studies at multi-megabar pressure and low temperature conditions, as well as condensed matter and materials studies using IR spectroscopy

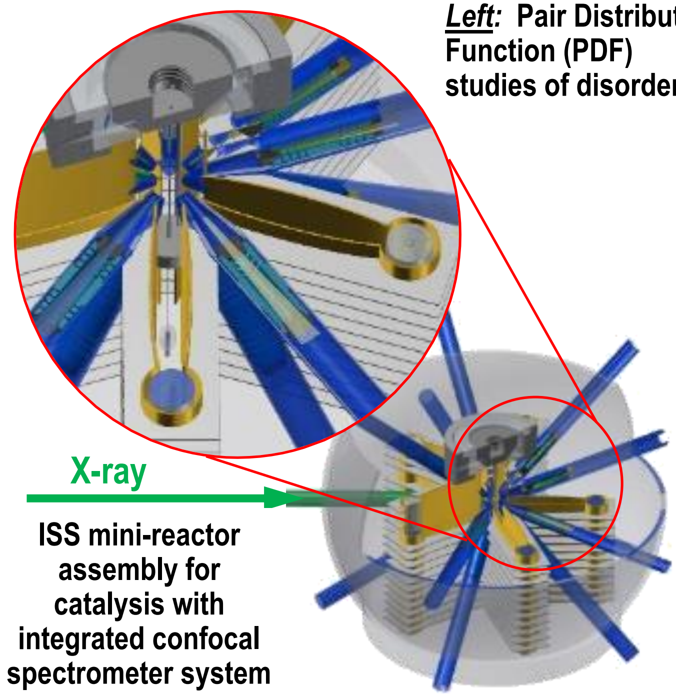






Top: XPD capillary sample cell allows *in-situ* studies of CO_2 capture in metal organic framework (MOF) structures

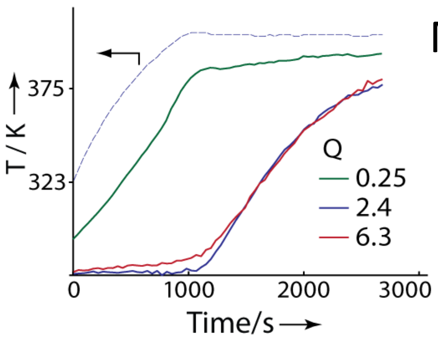
Left: Pair Distribution Function (PDF) studies of disorder



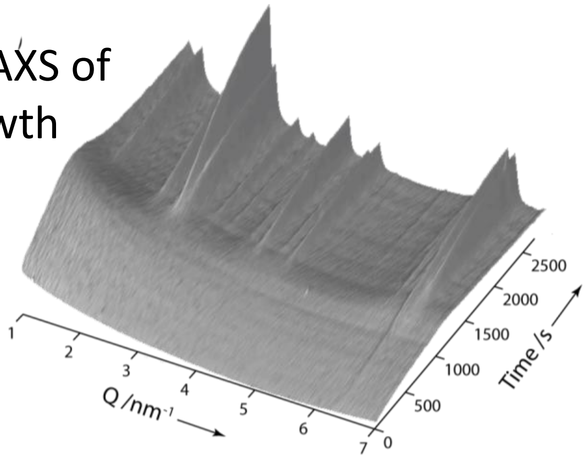
ISS mini-reactor assembly for catalysis with integrated confocal spectrometer system

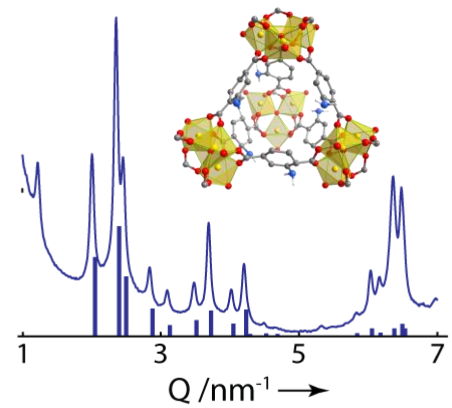
Substances: Synthesis, Functionalization & Testing

- Synchrotron radiation characterization methods available at NSLS-II delivers detailed information on the material synthesis mechanism



In situ WAXS of MOF growth





- Understanding of the material growth allows for optimization of synthesis parameters

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Substances: Synthesis, Functionalization & Testing

- X-ray diffraction helps tracking transformation of solids during adsorption – processes (CO_2/MOF)

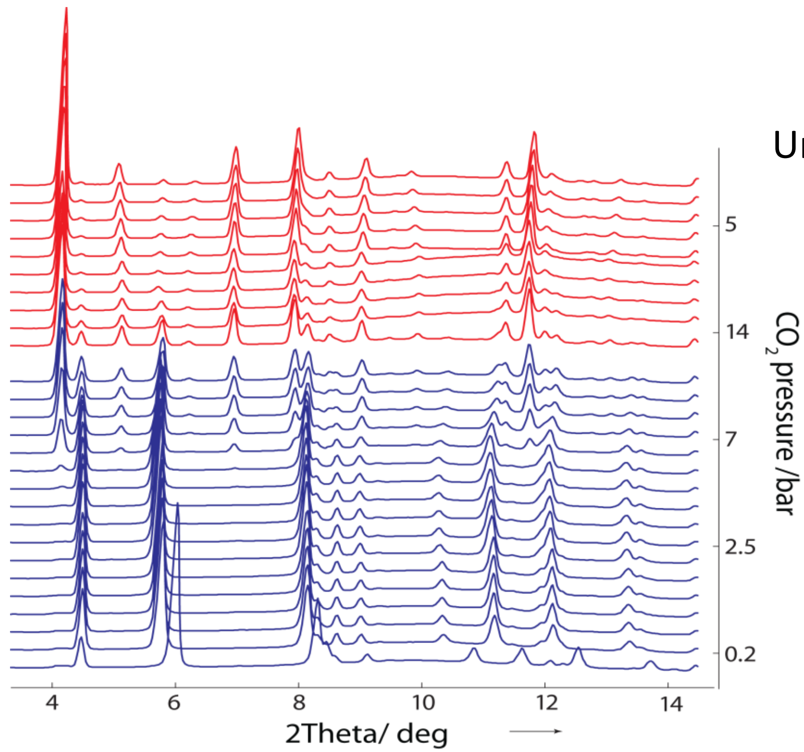
Structure

large pore

large/narrow pore

narrow pore

very narrow pore

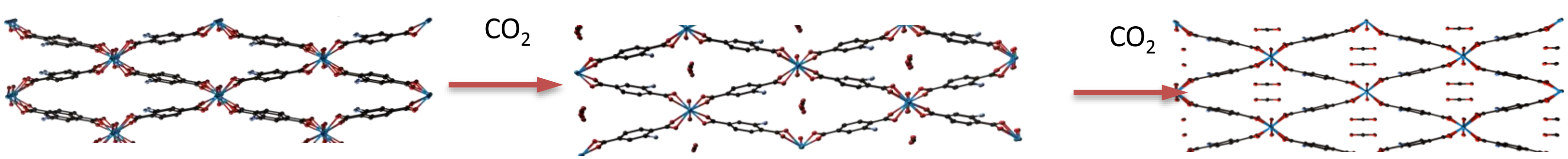


Unit cell volume, Å^3

1451

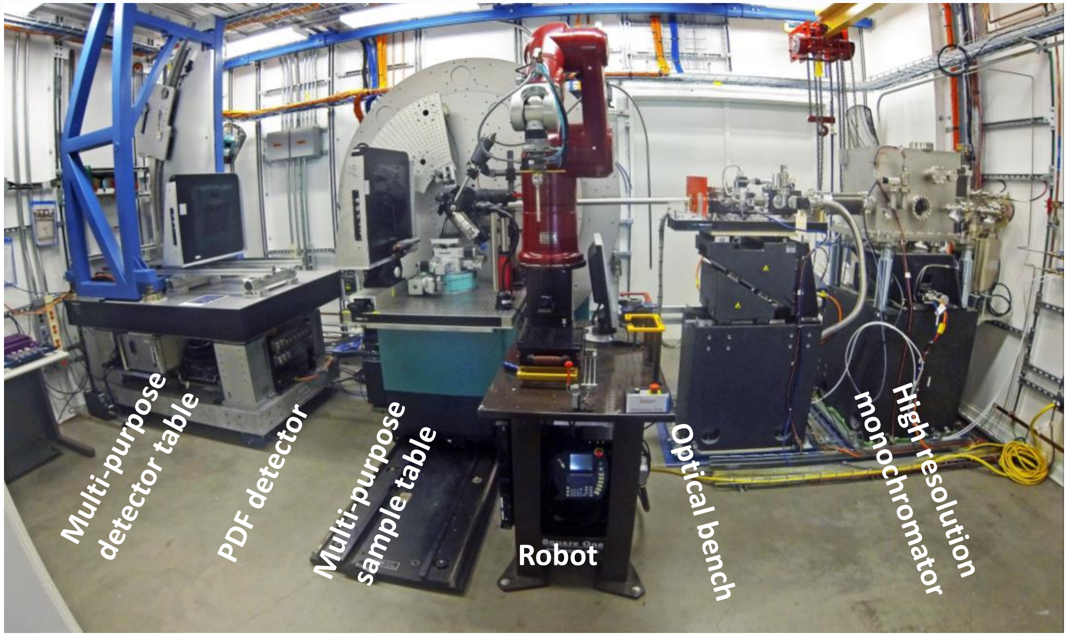
987

937



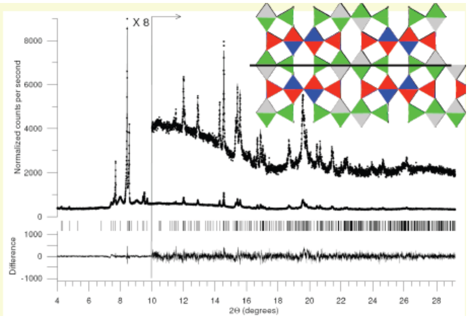
NSLS-II diffraction beamline (XPD)

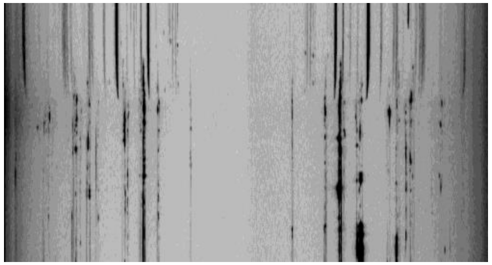
- Scientific capabilities
  - X-ray Powder Diffraction.
  - Pair Distribution Function Analysis (PDF).
  - Stress-Strain scanning.
  - Small Angle X-ray Scattering (SAXS).
  - Diffraction Tomography



Scientific priorities at XPD

1. CO_2 sequestration and catalysis. Conditions are as per down-hole: 300 Bar, 500ºC, flooded operation (sample wet at high T), high chloride concentrations (brine), down to pH=3, flammable gases (methane), sour gas capable (H_2S).
2. Structure of candidate H_2 storage material is to date determined *ex situ* on recovered structure. *In operando*, high resolution structure at high gradients (H_2 pressure, T) rather than on recovered samples, with time resolutions in the msecs (intermediate states in decomposition) and data at high Q to image individual particles to working device across length-scales µm-nm-Å.
3. Observe fuel synthesis AND in-situ high-Z material phase transformations up to 2000ºC while consuming trans-uranics with nitride fuels. Determine the local and long-range orders by combining real-space (PDF) and reciprocal-space (powder diffraction) methods, and cross-correlate with stress/strain mapping.





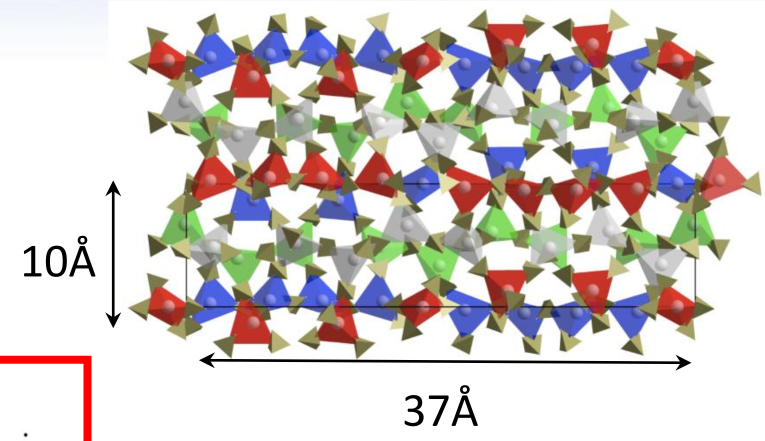
In situ High-Resolution Powder Diffraction

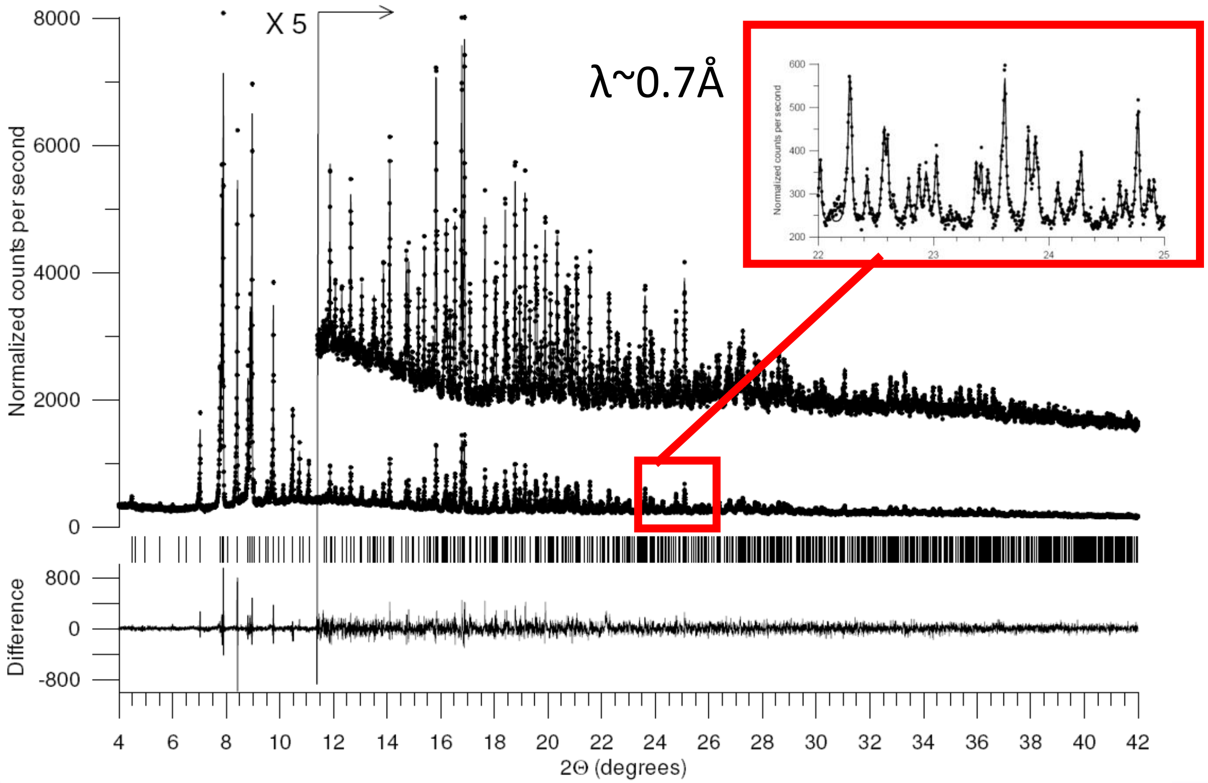
$M + B + 2H_2 \rightarrow MBH_4$ (M = Li, Na, ½Mg, etc)

550 - 700° C; 3 - 15 MPa (≈ 200 atm H2)

*Her et al (**2007**) Acta Crystallog., 63, 561*

"Low Temperature" phase of Mg(BH4)2

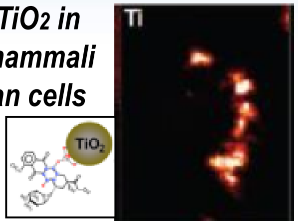


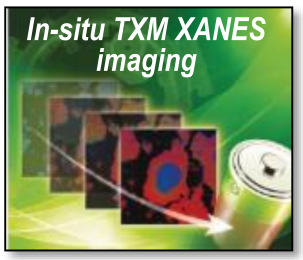


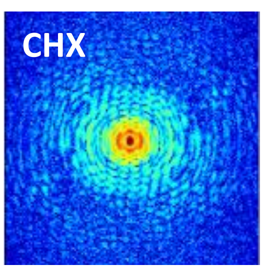
XPD is capable to reproduce this resolution and signal-to-noise discrimination from sample inside a HP vessel.

Nanoscale and Mesoscale Imaging

TiO_2 in mammalian cells





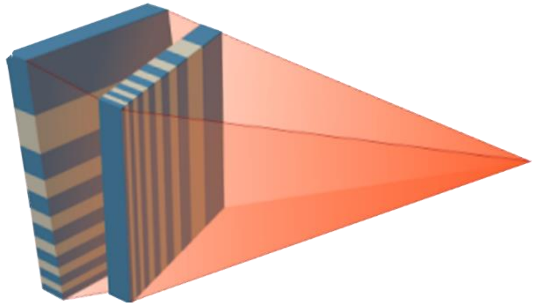


- Multi-scale In-situ Imaging of Heterogeneous Structures and Chemistry:
  - Hard X-ray Nanoprobe (HXN) – Structural and chemical imaging at 10 nm resolution, w/ ptychography to single-digit nm resolution
  - Submicron Resolution X-ray Spectroscopy (SRX) – High-throughput chemical imaging capability with sub-100 nm & sub-um resolution
  - X-ray Fluorescence Microscopy (XFM) – High-throughput mesoscopic scale imaging for elemental and chemical mapping
  - Full-field X-ray Imaging (FXI) – Hard X-ray TXM at 10-20 ms time-resolution & 30 nm spatial-resolution, and full 3D nano-tomography in <1 minute
  - Coherent Scattering (CHX and CSX-1) – coherent scattering and X-ray photon correlation spectroscopy with high coherent flux
  - Collectively this suite of dedicated imaging beamlines will enable a wide-range of *in-situ* structural and chemical studies of heterogeneous materials and biological systems

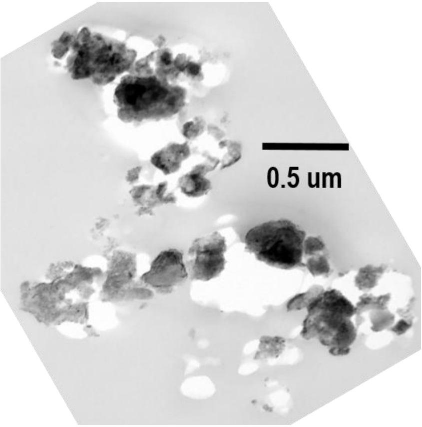
Hard X-ray Nanoprobe

- Ultra-high spatial resolution for hard x-rays
  - initial goal of ~10 nm and long-term goal of ~1 nm
- Advanced x-ray microscope
  - MLLs & ZP
  - ~1 nm positioning stability & ~2 nm/hour drift
  - simultaneous multi-modality imaging
- in-situ sample environment (T, liquid/gas)
  - High stability x-ray beam
  - High stability environment

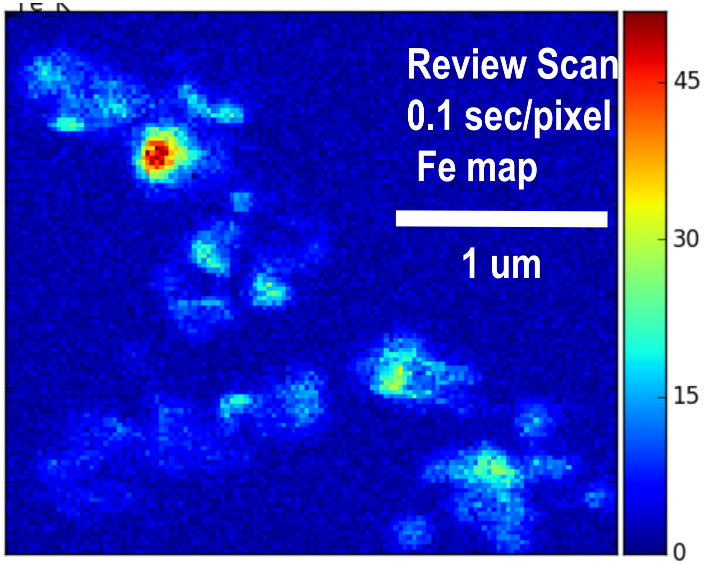
Multilayer Laue Lens (MLL)



SEM

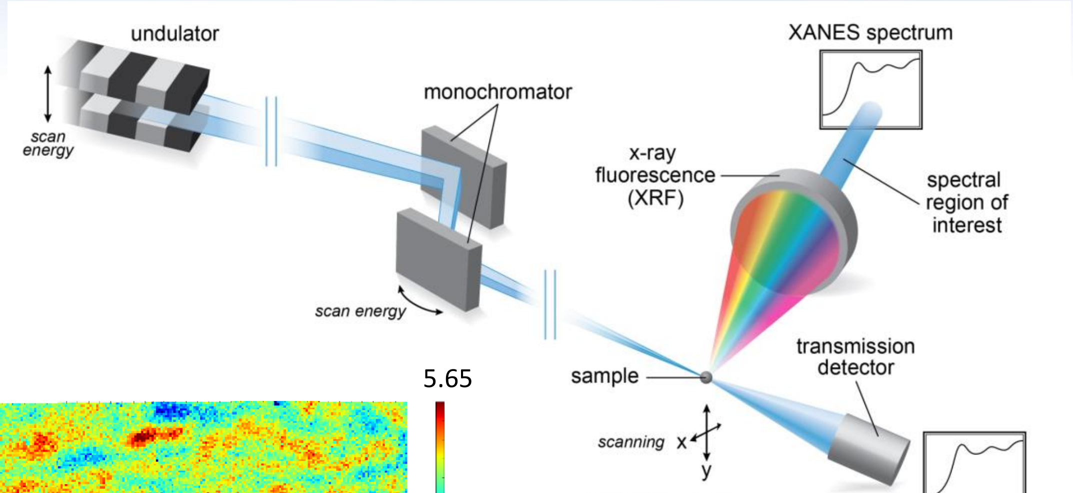


Elemental map

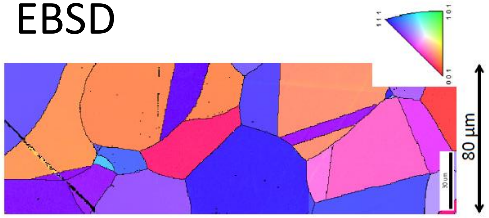


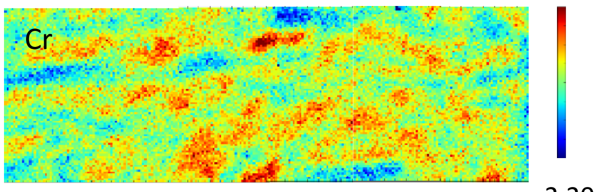
Submicron Resolution X-ray Spectroscopy

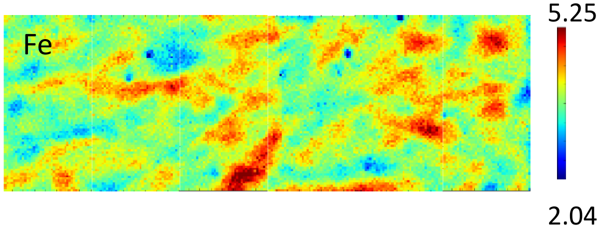
- High-throughput spectroscopic imaging capability (2D and 3D) with sub-100 nm spatial resolution



EBSD

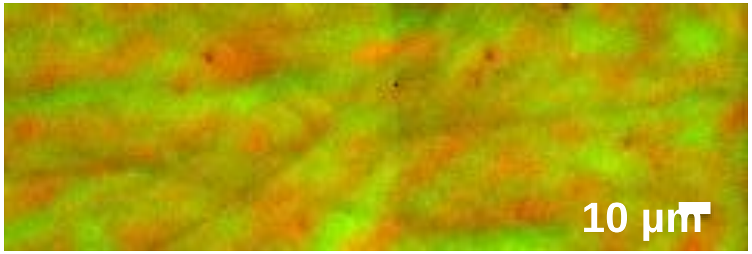






Cr

Fe



Overlay of Fe and Cr map shows segregation

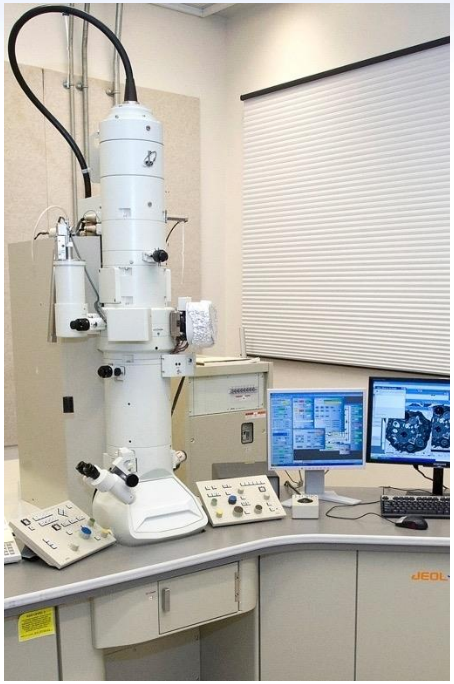
240 x 80 µm^2
Incident energy = 9 keV
Scan step size: 1 µm

- Stainless steel annealed at elevated temperature: phase segregation on the order of microns is detected by XRF

Center for Functional Nanomaterials

- A DOE National User Facility
- Nanoscience Research Center
- Seven facilities:
  - Materials Synthesis
  - Ultrafast spectroscopy
  - Nanofabrication
  - Proximal Probes
  - Theory & Computation
  - End Stations at the NSLS
  - Electron Microscopy
- www.bnl.gov/cfn

Electron Microscopy: Facilities at the CFN



JEOL 1400
Low voltage TEM

Soft & biological materials through cryo-microscopy

JEOL 2100F
Analytical electron microscope

Versatile analytical & in-situ experimentation

Hitachi HD2700C
STEM Probe corrector

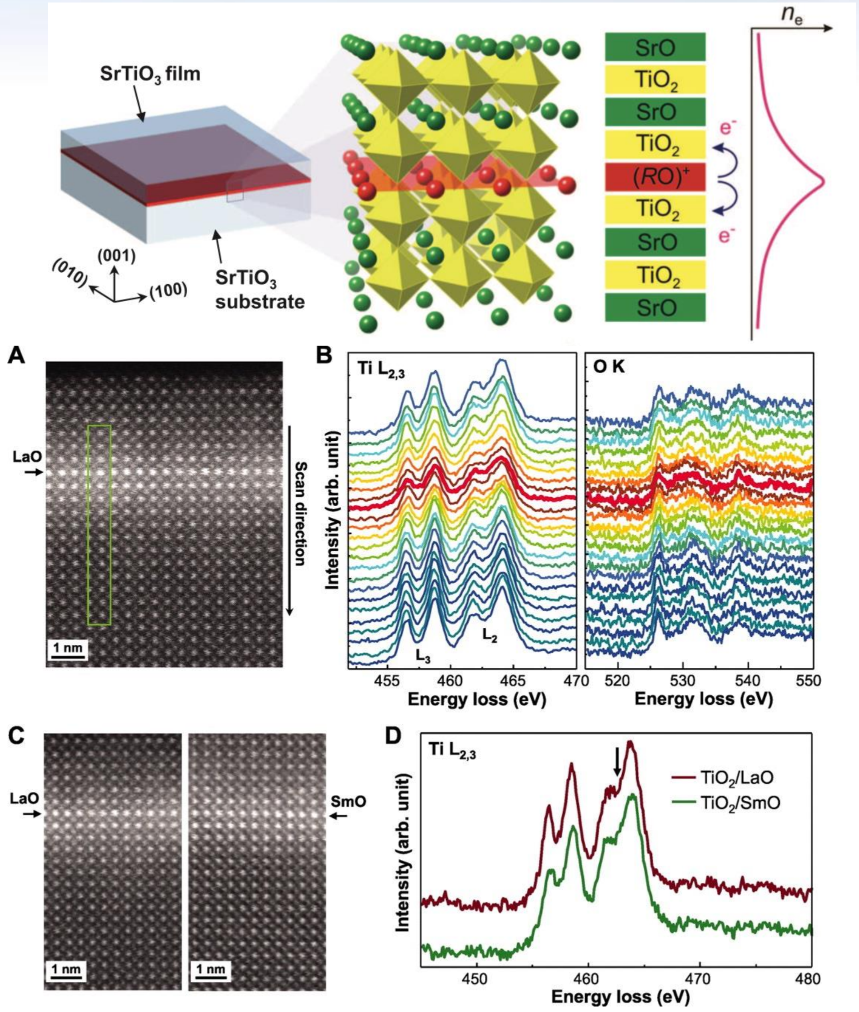
Forefront analytical instrument

Titan 80-300 - ETEM Image corrector

Environmental & in-situ studies

Hitachi HD2700C Analytical STEM

- Ultra-bright cold field emission source
- Spherical aberration corrector in probe forming optics
  - <1Å resolution
- Electron energy loss spectroscopy
  - 0.4 eV energy resolution
- Energy dispersive x-ray spectroscopy



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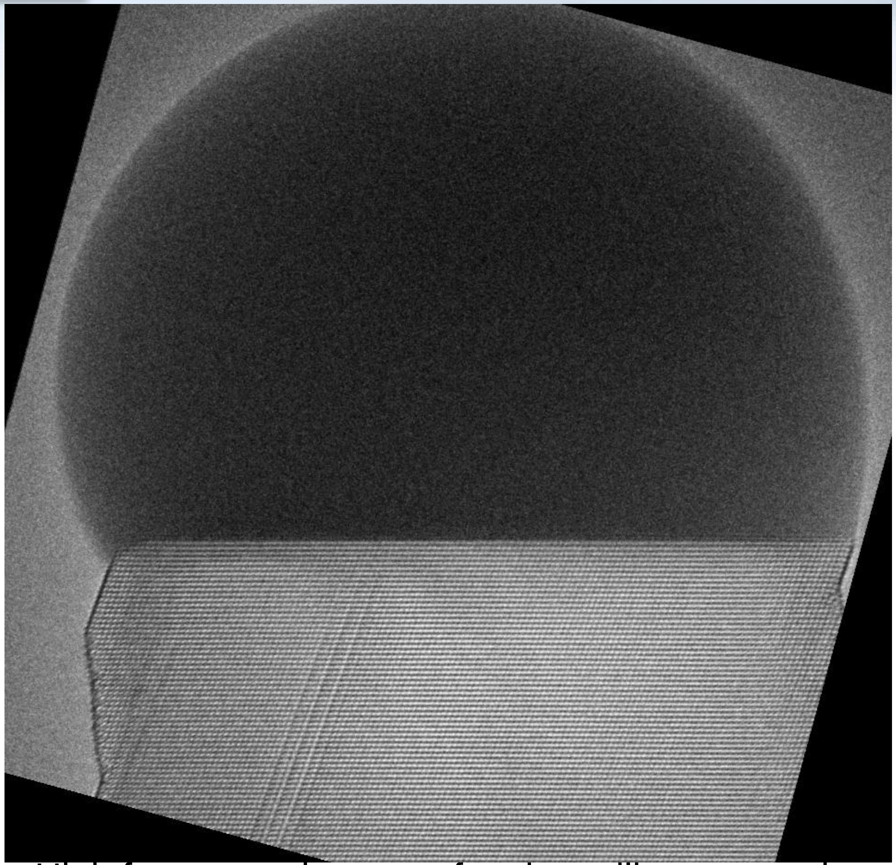
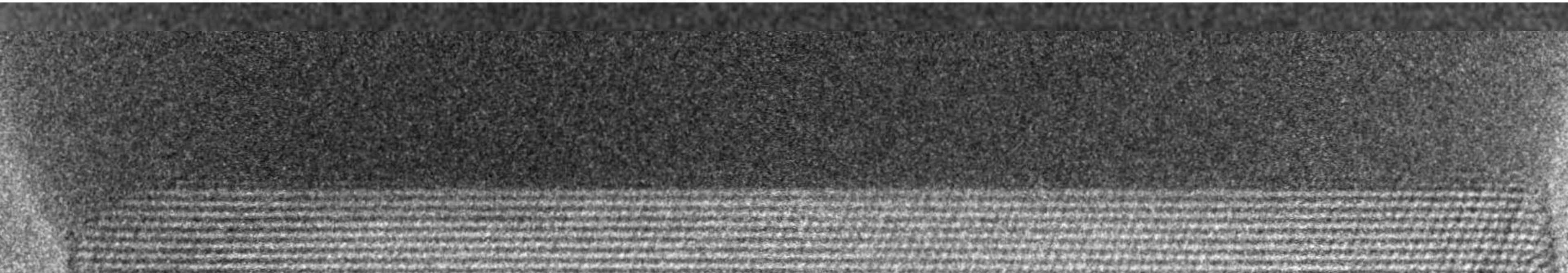
FEI Titan Environmental TEM/STEM

- Differential pumping apertures allow elevated gas pressures
  - up to 10 Torr
  - temperatures >1200C
- Spherical aberration correction
  - <1Å resolution
- Full analytical capabilities
- High frame rate imaging
- 400 frames/sec at 2k x 2k

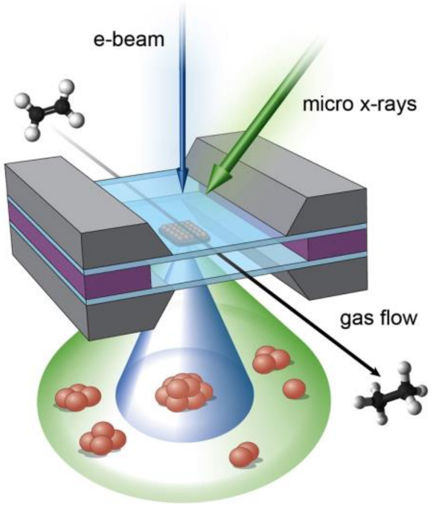


High frame rate images of carbon silicon nanowire growth (F. Ross @ IBM)



New instrument for "operando" research

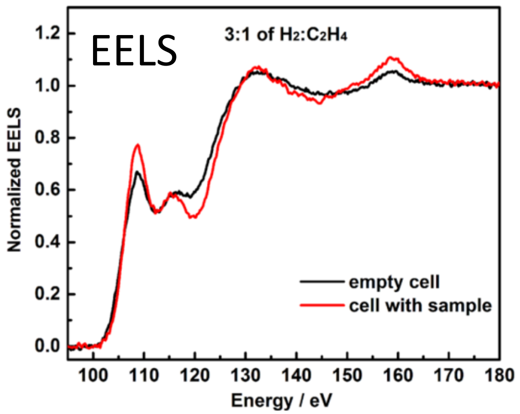
- New capabilities of analytical electron microscopy in 'real environments'
- Versatile STEM/TEM
- High-frame rate imaging
- Four integrated x-ray detectors for high-sensitivity chemical analysis
- Sample environments:
  - Heating
  - Liquid / electrochemistry
  - Atmospheric / catalysis

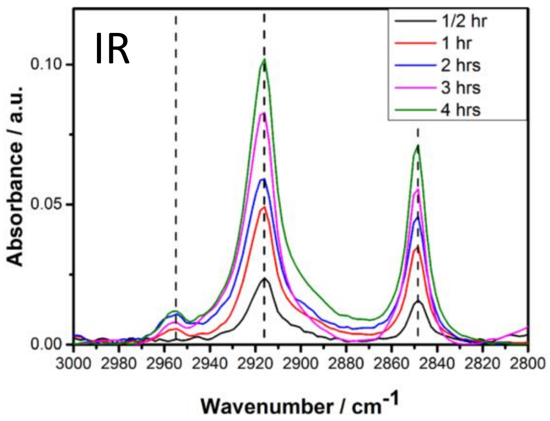


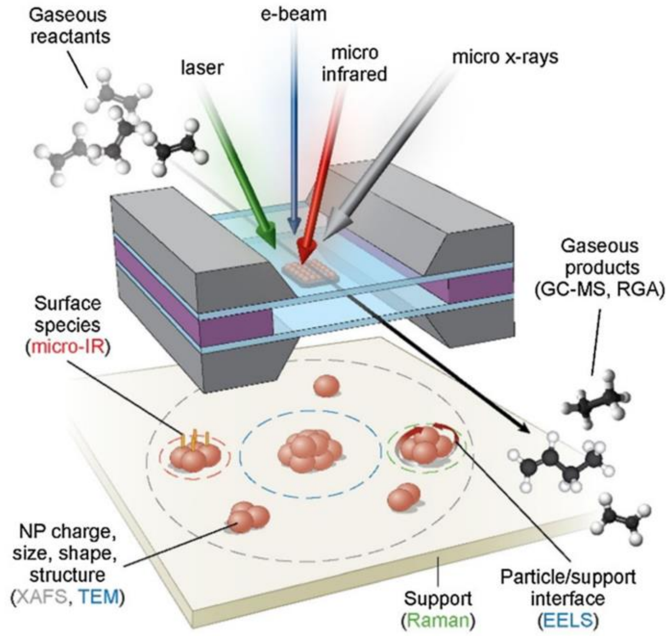
FEI Talos instrument

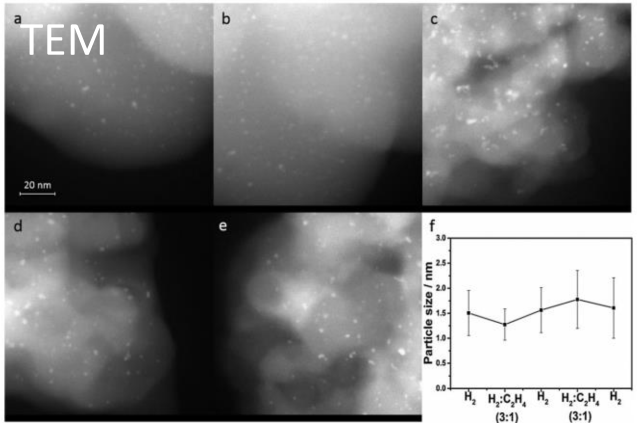
Integration of multiple techniques for in-situ research

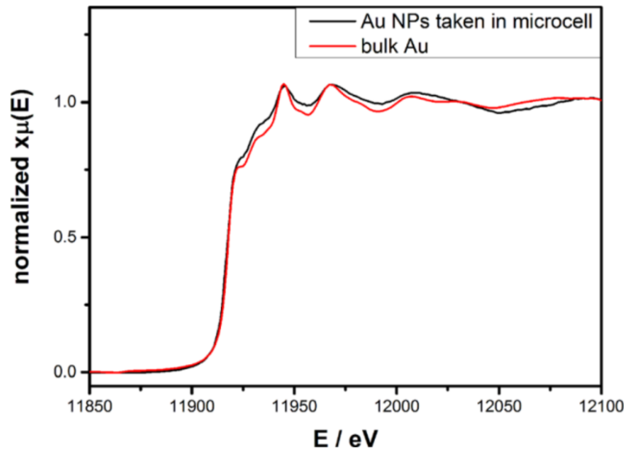
- Collective effort between NSLS-II, CFN and Chemistry department











Development of high throughput operando instrumentation

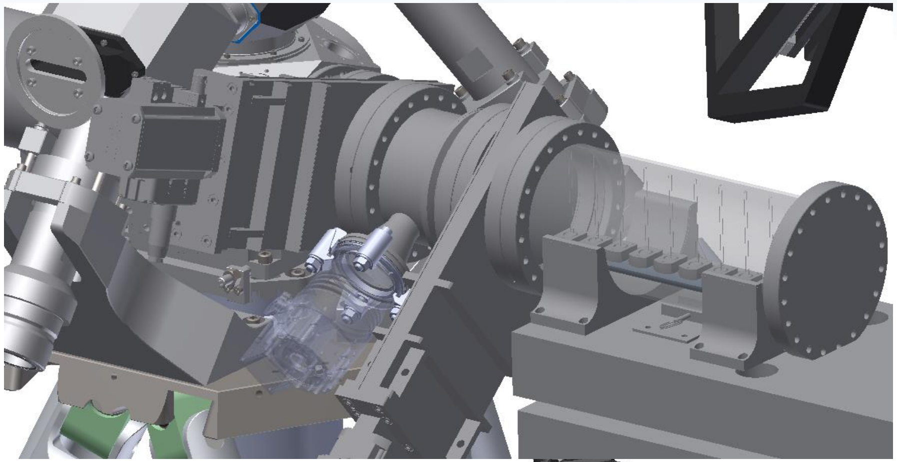
- Demand for high throughput analysis can be met with automation solutions

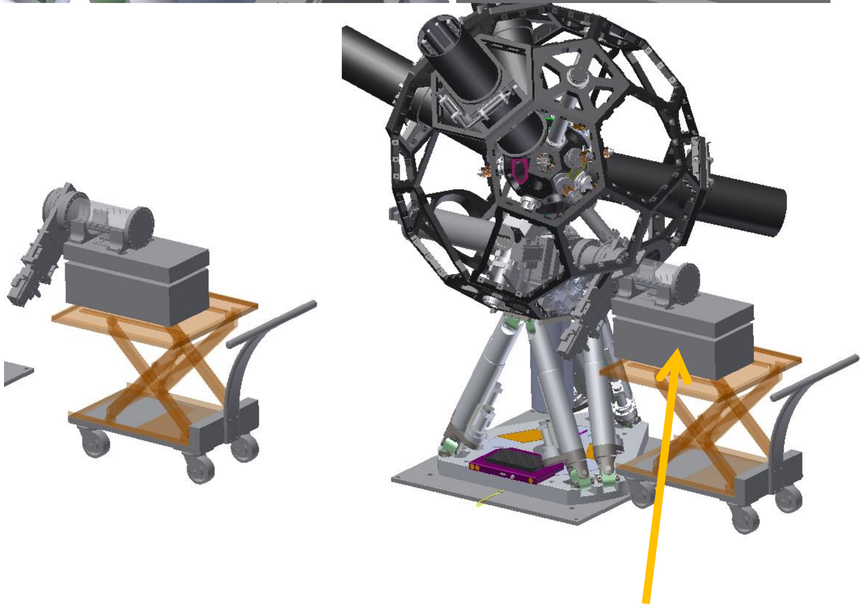
Robot sample handling system at NSLS-II beamline

- Sample environment systems allow to investigate functional systems under realistic industrial conditions (pressures/temperatures)

Gas delivery system at NSLS-II beamline

Sample transfer – from synthetic facility to characterization





Transport cart

- Sample transport and mounting
  - Sample transfer system with vacuum/inert gas compatible transfer box containing 10 samples
  - Containment can be shipped between facilities
- Double containment concept
- Automatic sample transfer
  - Samples are not exposed to air
  - Allows mail-in/remote access and high throughput
  - System includes bar-code encoding for samples and sample holders

The New York Center for Computational Sciences

- Joint venture between Stony Brook University's Institute for Advanced Computational Science and BNL Computational Science Center.
  - HPC Code Center is a central resource where domain scientists can bring their codes, or ideas for codes, and have those parallelized efficiently on High Performance Computing platforms.
  - New York Blue Supercomputer: centerpiece for the New York Center for Computational Sciences, which fosters research collaborations among research institutions, universities and companies throughout New York State.
  - IBM Blue Gene/Q for general purpose research that boasts 16 cores per compute node, links together 16384 processors, runs threaded code, has total peak performance of 200 teraflop, and placed fifth on the June 2012 Graph 500 benchmark list

Access modes

- GUP: general user proposals, valid to up to two years
- PUP: Partner user proposals, research groups making certain contributions and in return receiving a portion instrument time

- Get the instrument/method owner interested: provides access to constant, discretionary beamtime as well as high quality GUPs

Contact information

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  - National Synchrotron Light Source II

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  - 631 344 8641