Synchrotron Powder Diffraction at the Advanced Photon Source

Learn More About the Unique Capabilities for Synchrotron Powder Diffraction at the APS

The APS operates two dedicated powder diffraction beamlines (11-BM & 17-BM) described below. These instruments support a diverse scientific user community, focused on the structure-property relationships in inorganic oxides, porous materials, molecular systems, metallic alloys, and magnetic materials.

These start-of-the-art high energy probes enable experiments under ambient and in-situ conditions with exceptional angular and temporal resolution.

New user proposals for on-site & mail-in experiments are strongly encouraged; contact beamline staff for more information.

High-Resolution (11-BM)

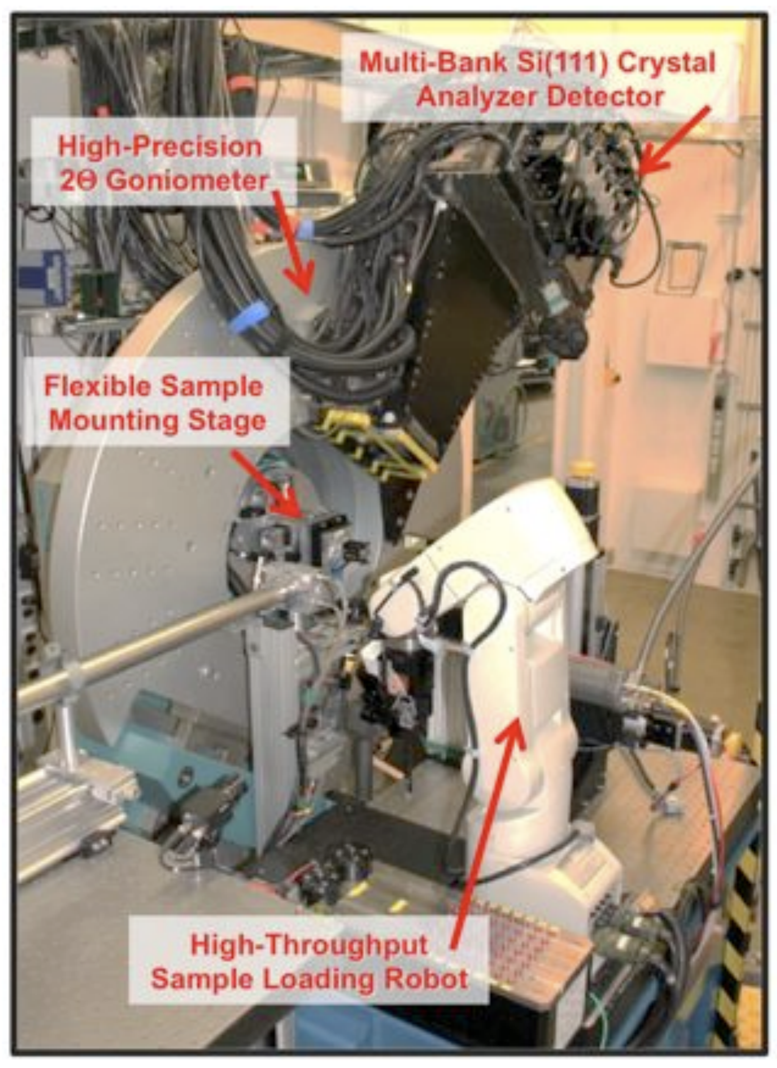
11-BM is a dedicated high resolution powder diffraction instrument at the APS. It affords powder diffraction data with world-class resolution and outstanding sensitivity. The beamline supports both on-site and rapid access mail-in user programs.

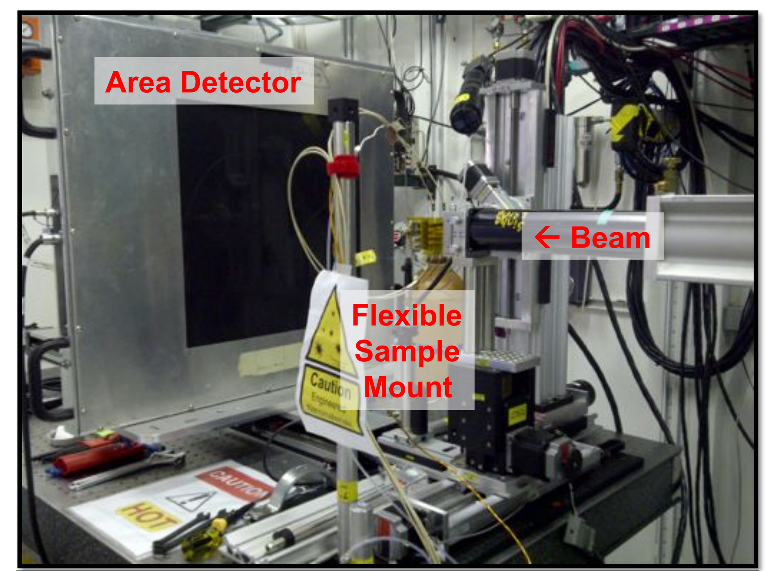
Instrument Specifications

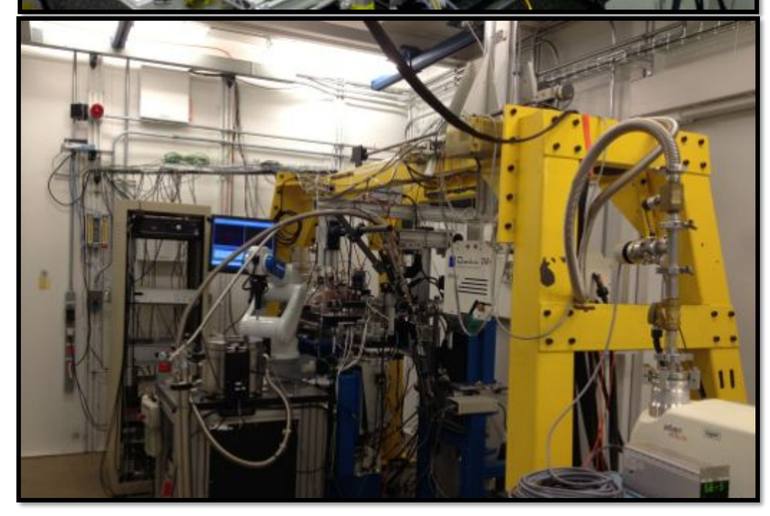
- ✧ Energy Range: 15 – 35 keV
- ✧ Resolution: $\Delta d/d \approx 2\times10^{-4}$
- ✧ Scan Time: 10 - 60 minutes

Sample Environments

- ✧ Oxford Cryostream (100 - 450 K)
- ✧ Cyberstar Hot Gas Blower (25 - 900 °C)
- ✧ Helium Flow Cryostat (5 - 150 K)
- ✧ Gas Reaction Cell (under development)
- ✧ High Temp (>1000 °C) furnace (planned)







Fast Area Detector (17-BM)*

17-BM is a powder diffraction instrument at the APS optimized for in-situ experiments under non-ambient conditions. A state-of-the-art area detector enables fast data acquisition, and on-site users benefit from a wide variety of flexible sample environments.

Instrument Specifications

- ✧ Energy Range: 7 – 20 keV
- ✧ Resolution: $\Delta d/d \approx 1\times10^{-3}$
- ✧ Scan Time: 0.1 - 60 seconds

Sample Environments

- ✧ Flowcell Reaction Furnace (<900 °C)[4]
- ✧ Oxford Cryostream (100 - 450 K)
- ✧ In-Situ Controlled Atmospheres
- ✧ Diamond Anvil Cells (<10 GPa)

** Formerly 1-BM; moved to 17-BM Fall 2012*

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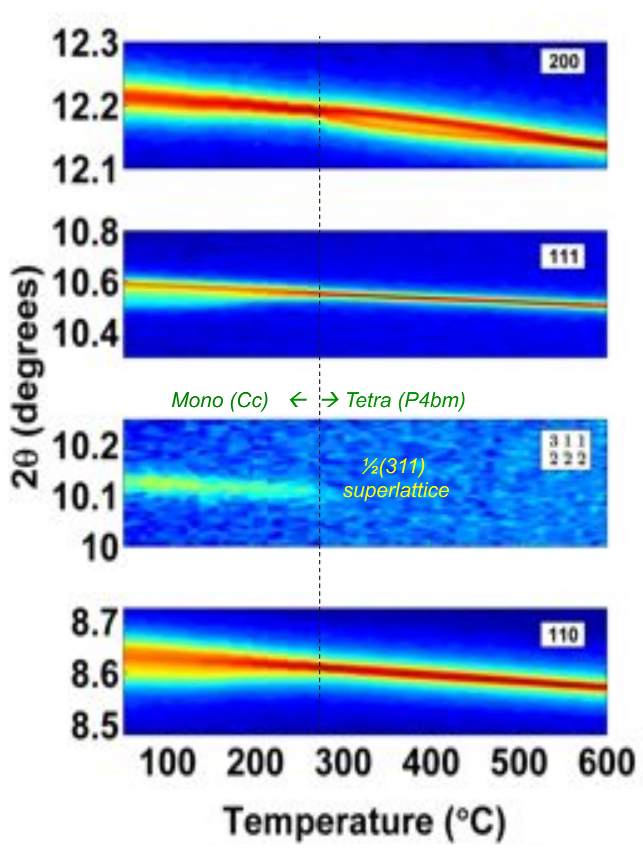
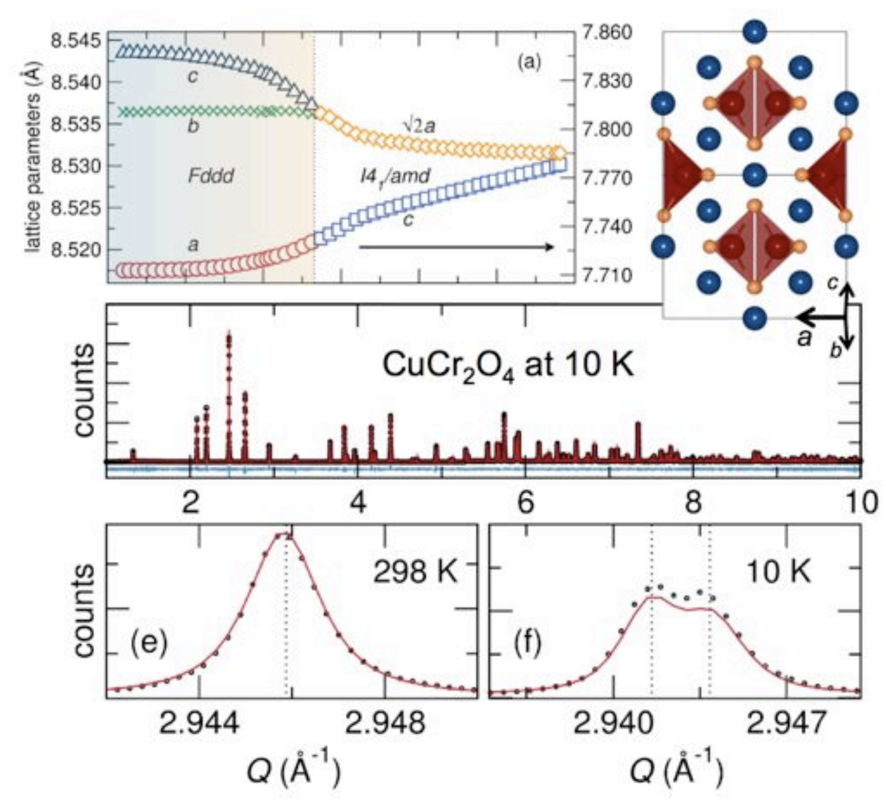
11-BM Highlight : Non-Ambient High-Resolution Diffraction



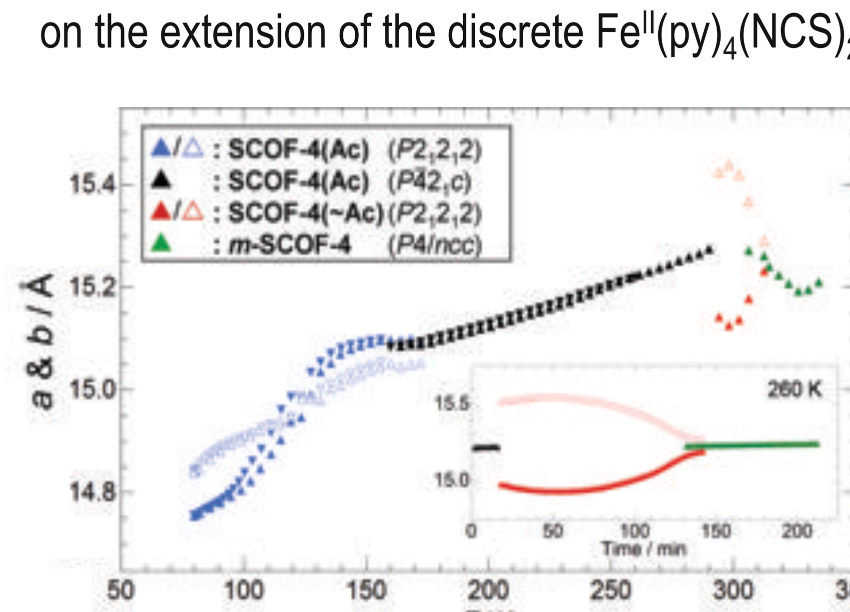
In-situ high temperature pXRD shows structural origins of depoling hysteresis in $Na_{0.5}Bi_{0.5}TiO_3$ piezoelectrics, and reveals new complexity in the parent ceramic material at ambient temperatures.[2]

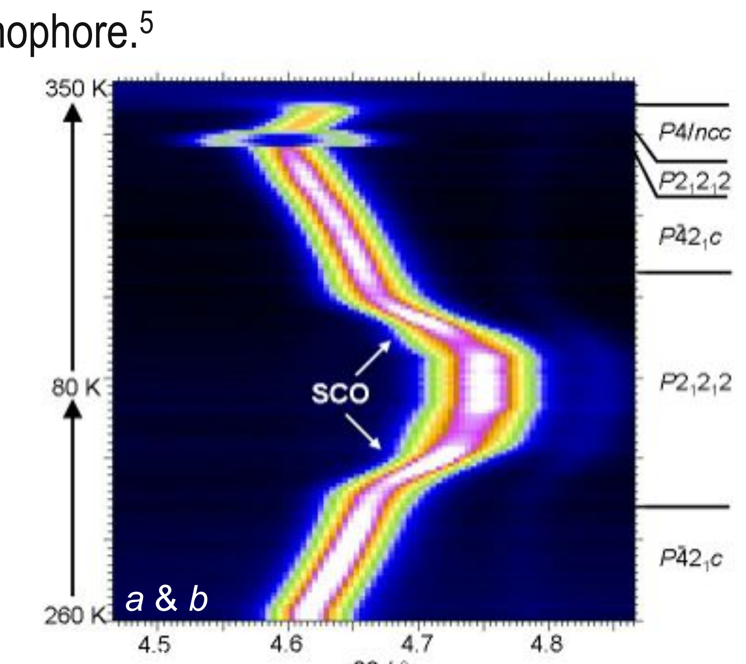
Cryogenic measurements at 11-BM help elucidate coupling between spin, lattice, and orbital ordering in spinel metal oxides. High-resolution data is required to observe the subtle low temperature structural distortions.[1]



17-BM Highlight : In-Situ Studies of Multi-Functional Materials

The co-existence of nanoporosity and electronic switching in molecular framework materials promises an array of unprecedented physiochemical properties. Such solids are unique in allowing detailed in situ studies of the steric and electronic influence of adsorbed guests on the spin crossover (SCO) centres. **SCOF-4·(Ac)** ($Fe(1,2\text{-bis}(4\text{-pyridyl})ethane)_2(NCS)_2\cdot3(acetone)$) represents a targeted variation in a series of Spin Crossover Framework (SCOF) materials based on the extension of the discrete $Fe^{II}(py)_4(NCS)_2$ chromophore.[5]





In-situ diffraction experiments at 1-BM revealed nine distinct structural phases within a complex sequence of phase transitions that describe both the SCO and the host-guest properties of **SCOF-4**.

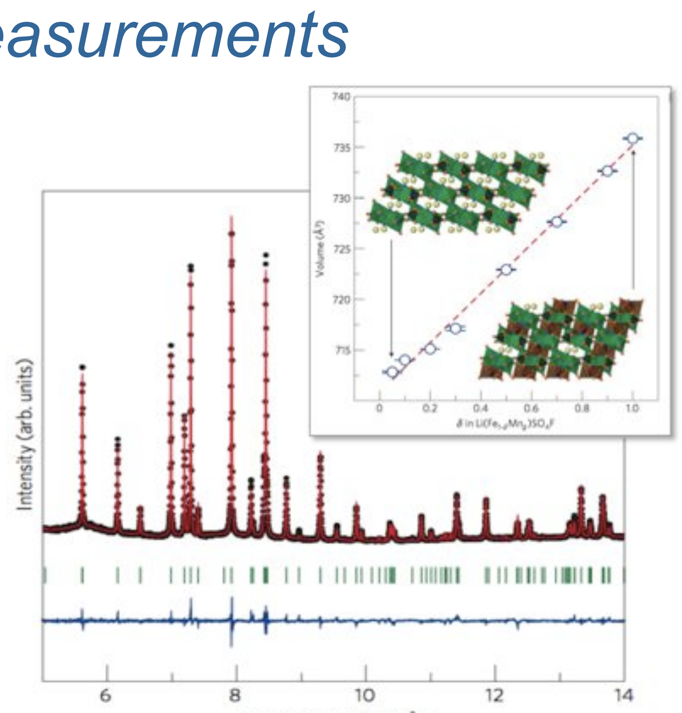
Rapid Access Mail-In Measurements

11-BM offers a unique rapid-access mode for mail-in access to high-resolution powder diffraction data. Users are notified when data are available for electronic download. This free service for non-proprietary users is open to both US and international research teams.

- ✧ Short Proposal, Fast Review
- ✧ Limited to 8 Hours per Proposal
- ✧ Free for Non-Proprietary Work
- ✧ Sample Mounting Kits Provided
- ✧ Scans at Ambient & 100-450 K
- ✧ Fixed Energy 30 keV (0.41 Å)
- ✧ Download Data in ~ 3 weeks*

** typical, subject to beamline scheduling*

more information
http://11bm.xray.aps.anl.gov
or email: 11bm@aps.anl.gov



Data collected via the 11-BM mail-in program helped explain electrochemical structural relationships in Li-ion battery electrodes. High resolution patterns allowed a precise determination of small (~0.5%) volume changes with Li-insertion.[3]

Proposals for On-Site Experiments

Perform your next experiment here at the APS !!

Beamlines 17-BM & 11-BM both currently accept user proposals for on-site experiments. Users may submit their proposal via the APS webpage for any of the 3 proposal deadlines each calendar year. Successful proposals are awarded beamtime in the next APS run cycle.

APS PROPOSAL DEADLINES:
March / July / October

for details & exact dates see:
http://www.aps.anl.gov/Users/Calendars/

Advantages of On-Site Synchrotron Experiments

- ✧ Access Full Suite of Beamline In-Situ Environments
- ✧ Design, Build, and Bring your Custom Sample Cell
- ✧ Real-Time Experimental Feedback & Interaction
- ✧ Extensive Support from Experienced Beamline Staff
- ✧ Perform Detailed Multi-day Parametric Studies

New User Proposals Are Always Encouraged… Questions? Get in Touch!

References:

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(4) Chupas, P. J.; Chapman, K. W.; Kurtz, C.; Hanson, J. C.; Lee, P. L.; Grey, C. P., *J. Appl. Crystallogr.* **2008**, *41*, 822.
(5) Halder, G.J.; Chapman, K.W.; *et al.*, *J. Am. Chem. Soc.* **2008**, *130*, 17552

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