X-RAY ATOMIC PAIR DISTRIBUTION FUNCTION (PDF) BEAMLINE, XPD-2

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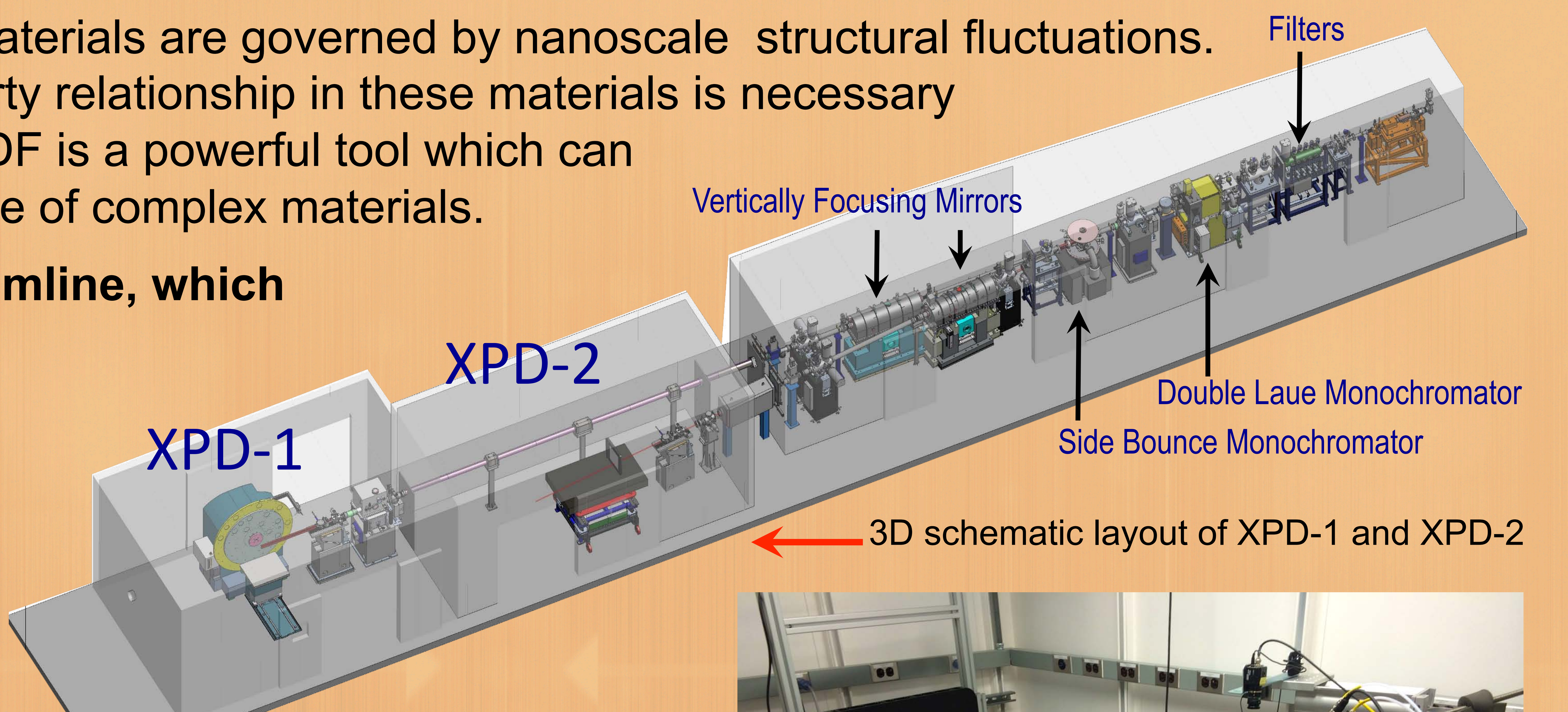
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

SCIENTIFIC SCOPE

The properties of most technologically important materials are governed by nanoscale structural fluctuations. Therefore, understanding the local structure-property relationship in these materials is necessary to manipulate and optimize their properties. The PDF is a powerful tool which can be used to study local atomic ordering in wide range of complex materials.

XPD-2 will be a dedicated general user PDF beamline, which uses extremely high flux X-rays from a 68-pole damping wiggler at the the NSLS II. XPD-2 enables PDF, SAXS, and WAX measurements in a wide variety of sample environments.



3D schematic layout of XPD-1 and XPD-2

TECHNICAL CAPABILITIES

<u>Techniques:</u> High-throughput PDF, SAXS, WAXS

<u>Available energies</u>: 39 keV, 64 keV & 75 keV

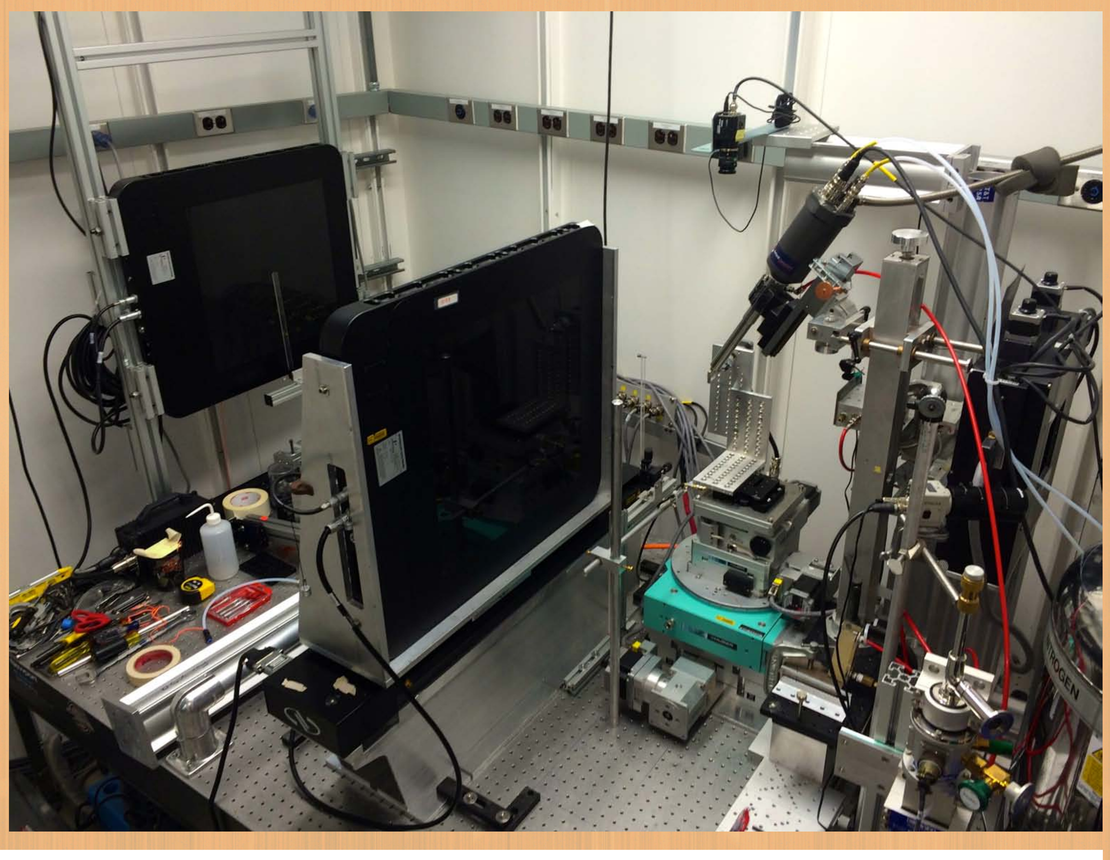
<u>Energy resolution</u>: 10^{-2} to 2×10^{-3} ΔE/E

<u>Time resolution</u>: <1s (dynamics, rapid acquisition)

<u>Sample environments:</u> (5-500) K cryostat, (80-500) K cryostream, flow-cell with gas handling capabilities, chemical reaction cells, magnetic/electric fields.

<u>Software</u>: EPICS, QXRD, Fit2D, PDFgetX3, SrFit, PDFgui, GSAS I & II, FullProf, Python.

Prototype end-station for XPD-2 X17A at NSLS.

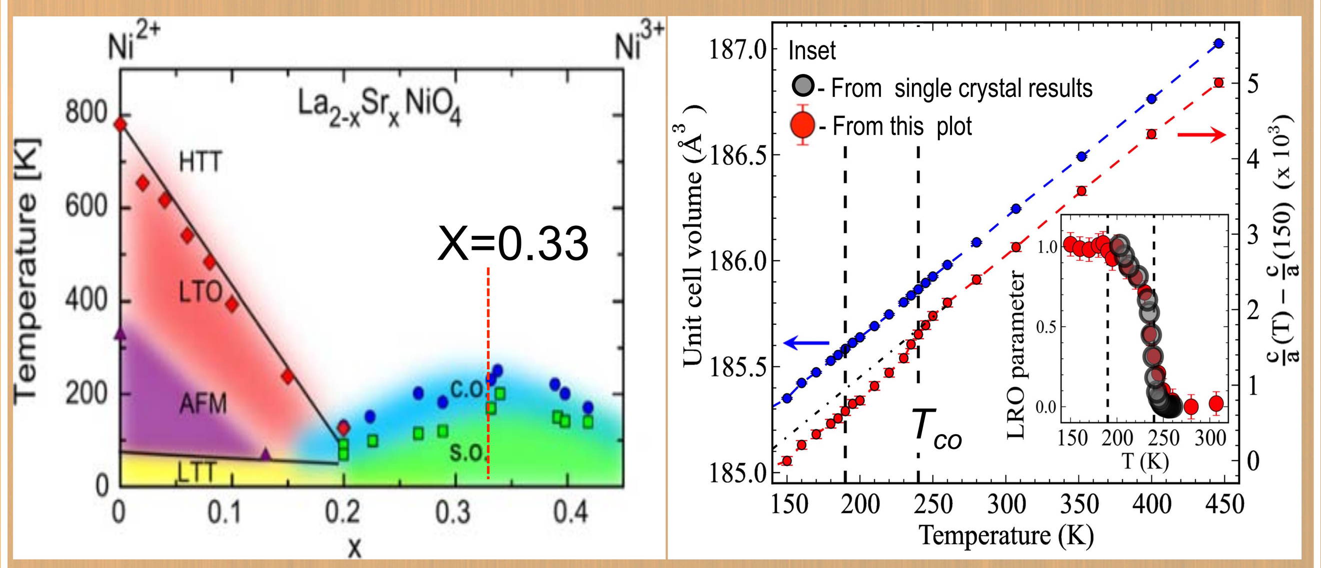


APPLICATIONS

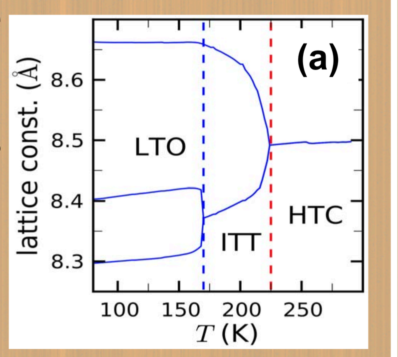
Strongly correlated materials

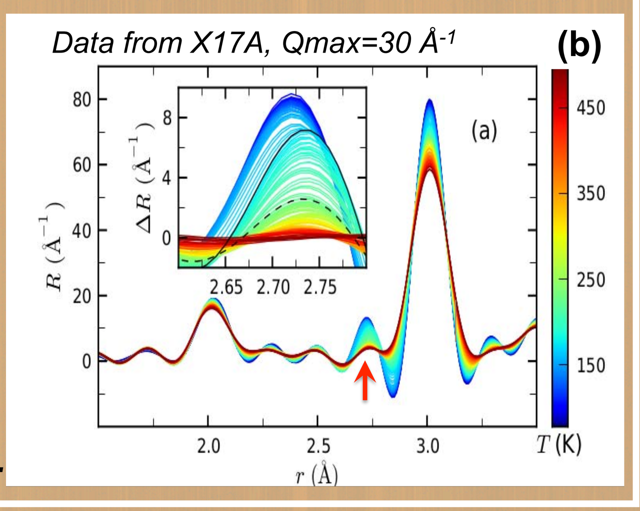
High-throughput, high r-resolution measurements at XPD-2 will enable average and local structural studies of the <u>full phase diagrams</u> of strongly correlated systems giving key insights into the structure-property relationship.

Ex. 1: $La_{2-x}Sr_xNiO_4$ (LSNO) shows robust charge order below T_{co} = 240 K. Figures show the phase diagram of LSNO and the T-evolution of lattice constants at x=0.33 (*Abeykoon et. al. Phys. Rev. Lett.* **111**, *096404*). Rapid data acquisition times at XPD-2 will give the opportunity to investigate the T-evolution of both local & average structures of LSNO over its full composition range.



Ex. 2: <u>PDF is sensitive to the local structural response to electronic states of materials.</u> Two-stage metal insulator phase transition occurs in $LiRh_2O_4$ with known Rh^{4+}-Rh^{4+} dimers in the low-T phase (LTO). For the first time, high r-resolution PDF studies confirmed the existence of dimers in all three phases. T-evolution of lattice constants and the dimer distance RDF peak (↑) are shown in figures **(a)** & **(b)** respectively. Inset of **(b)** displays differential RDF at the dimer distance obtained by subtracting a reference RDF(500 K) from each RDF. Black lines at 170 K (solid), & 225 K (dashed) denotes differential RDFs at phase transition temperatures. *Knox et. al. Phys. Rev. B 88, 174114 (2013).*

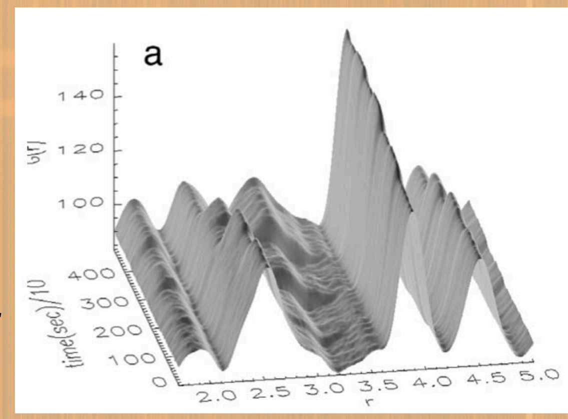




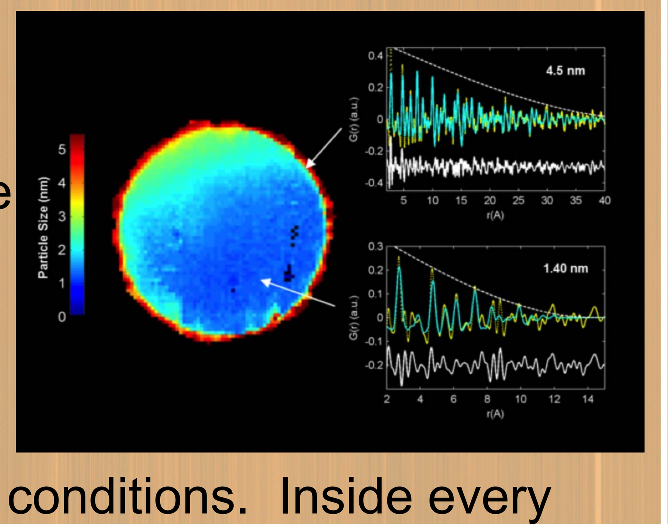
Catalysts

XPD-2 provides high flux and rapid acquisition (<1s) necessary for the <u>in-situ characterization of catalysts</u>.

Ex. 1: Time resolved series of PDF's taken during pulse sequence of $CO/He/O_2$ /He over fully reduced CeO_2/Cu catalyst at 200°C. *Hanson et al., Catalysis Today 229 (2014) 64*



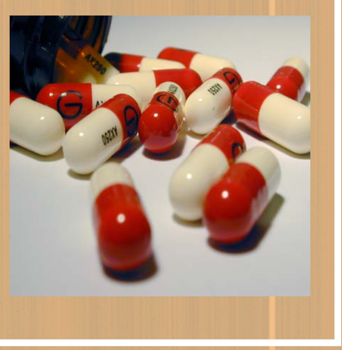
Ex. 2: Local structures of a real device, Pd/Al_2O_3 industrial catalyst body, studied by <u>PDF tomography (ct-PDF)</u>. Figure shows distribution of particle sizes of fcc Pd within the 3 mm Φ catalyst body under reducing conditions. Inside every pixel is a complete PDF that can be fitted with models for quantitative nanostructure information. *Jacques et al., Nat. Commun.* **4**, *2536 (2013)*



Pharmaceuticals

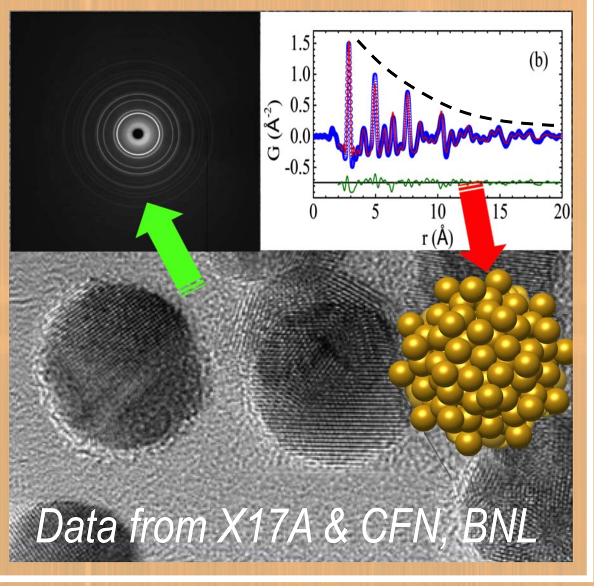
High flux at XPD-2 will ensure excellent PDF data from <u>low scattering materials</u>.

Ex. 1: Fingerprinting and structural studies of drugs can largely benefit from this.



Nanomaterials

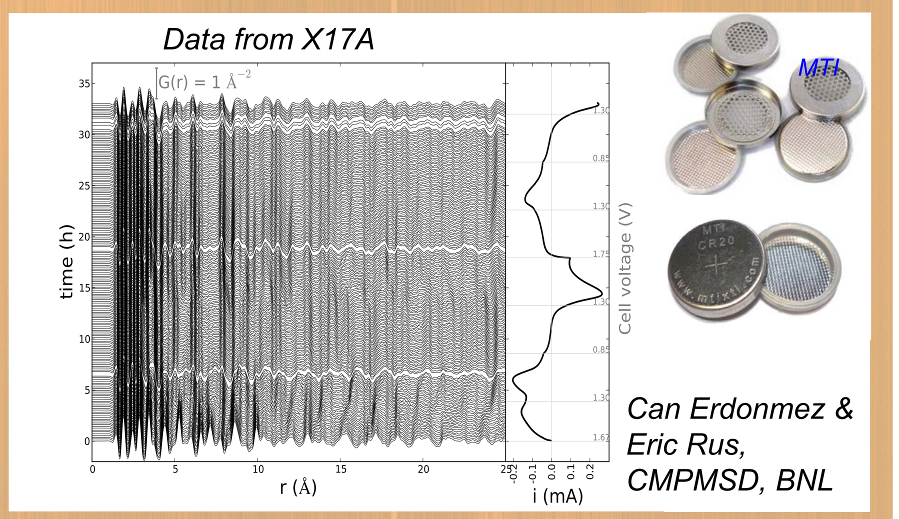
High flux and high energy at XPD-2 will ensure excellent PDF data from nanomaterials up to high Q required for high r-space resolution. Potential user programs between the CFN & XPD-2 will provide complementary TEM/ePDF data.



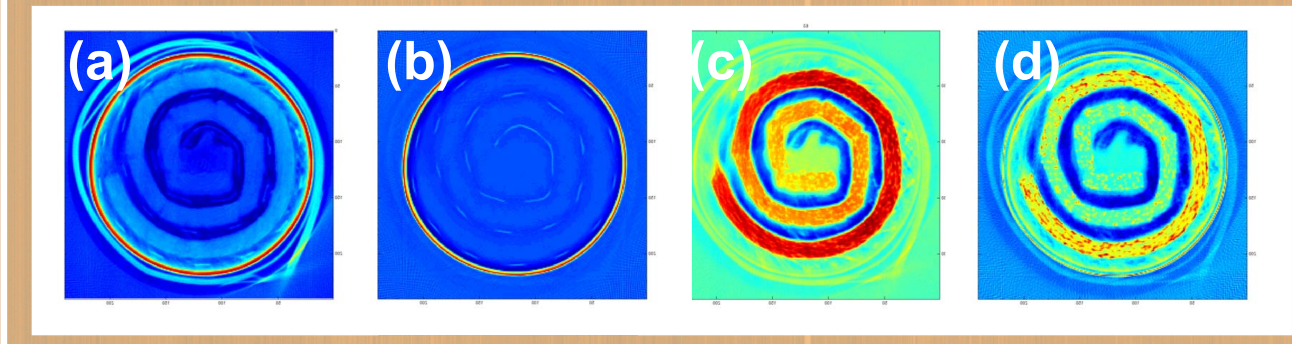
Batteries

Combining PDF with voltage-current measurements can give key insights into <u>local structural changes in batteries during charging/discharging cycles</u>. The spatially resolved <u>XRD and PDFs (tomography) can be used to probe deep inside NiMH batteries</u>.

Ex. 1: Figure shows PDFs and a V-I plot of an Alkaline battery during charging and discharging cycles



Ex. 2: XRD-ct of a NiHM battery



(a) XRD-ct image from high-Q region (mostly Compton scattering) which scales with number of electrons. Image shows the steel case (red), and the Kapton tube supporting the battery (outer ghostly image). **(b)** XRD-ct image from a steel peak. **(c), (d)** Images from peaks in the cathode XRD. The cathode (spiral wound with a separator) and a current collector between the jelly-rolls are seen on these images. *Marco DiMichiel et. al. ESRF*