2011 Neutron/X-ray Scattering Experiments and Descriptions

Week 1, Neutron experiments

Group	June 15th	June 16th	June 17th
A	**N1.** Triple-Axis (HB-1A)	**N11.** VULCAN (BL-7)	**N5.** GP SANS (CG-2)
B	**N1.** Triple-Axis (HB-1)	**N10.** SNAP (BL-3)	**N6.** Bio-SANS (CG-3)
C	**N1.** Triple-Axis (HB-3)	**N3.** POWGEN (BL-11A)	**N7.** EQ-SANS (BL-6)
D	**N8.** BASIS (BL-2)	**N4.** SC Diff. (HB-3A)	**N13.** Liq. Refl. (BL-4B)
E	**N9.** SEQUOIA (BL-17)	**N2.** Powder Diff. (HB-2A)	**N12.** Magn. Refl (BL-4A)
F	**N2.** Powder Diff. (HB-2A)	**N6.** Bio-SANS (CG-3)	**N9.** SEQUOIA (BL-17)
G	**N4.** SC Diff. (HB-3A)	**N5.** GP SANS (CG-2)	**N8.** BASIS (BL-2)
H	**N3.** POWGEN (BL-11A)	**N7.** EQ-SANS (BL-6)	**N1.** Triple-Axis (HB-3)
I	**N10.** SNAP (BL-3)	**N13.** Liq. Refl. (BL-4B)	**N1.** Triple-Axis (HB-1)
J	**N11.** VULCAN (BL-7)	**N12.** Magn. Refl (BL-4A)	**N1.** Triple-Axis (HB-1A)
K	**N5.** GP SANS (CG-2)	**N9.** SEQUOIA (BL-17)	**N3.** POWGEN (BL-11A)
L	**N6.** Bio-SANS (CG-3)	**N8.** BASIS (BL-2)	**N10.** SNAP (BL-3)
M	**N13.** Liq. Refl. (BL-4B)	**N1.** Triple-Axis (HB-3)	**N11.** VULCAN (BL-7)
N	**N7.** EQ-SANS (BL-6)	**N1.** Triple-Axis (HB-1)	**N2.** Powder Diff. (HB-2A)
O	**N12.** Magn. Refl (BL-4A)	**N1.** Triple-Axis (HB-1A)	**N4.** SC Diff. (HB-3A)

HFIR experiment
SNS experiment

Week 2, X-Ray experiments

Group	June 20th	June 22th	June 23th	June 24th
A	**X2**, High Energy Stress-Strain	**X14**, SAXS	**X15**, Tomography	**X2**, High Energy Stress-Strain
B	**X5**, Inelastic X-ray	**X5**, Inelastic X-ray	**X18**, SAXS-Bio	**X15**, Tomography
C1	**X20**, nano-probe	**X19**, EXAFS	**X17**, High Pressure	**X17**, High Pressure
C2	**X4**, μ-flourescence Imaging	**X19**, EXAFS	**X17**, High Pressure	**X17**, High Pressure
D	**X2**, High Energy Stress-Strain	**X10**, Time-resolved Diff.	**X16**, USAXS	**X2**, High Energy Stress-Strain
E	**X21**, Transmission x-ray microscopy	**X12**, High Resolution Diff.	**X12**, High Resolution Diff.	**X7**, XMCD-Hard
F	**X21**, Transmission x-ray microscopy	**X13**, PDF	**X13**, PDF	**X18**, SAXS-Bio
G	**X3**, Tomography	**X1**, Powder Diff.	**X22**, GIXS	**X11**, GISAXS
I	**X10**, Time-resolved Diff.	**X16**, USAXS	**X7**, XMCD-Hard	**X21**, Transmission x-ray microscopy
J	**X20**, nano-probe	**X6**, XMCD-Soft	**X8**, SAXS	**X22**, GIXS
L	**X14**, SAXS	**X13**, PDF	**X13**, PDF	**X3**, Tomography
M	**X19**, EXAFS	**X8**, SAXS	**X17**, High Pressure	**X17**, High Pressure
N	**X4**, μ-flourescence Imaging	**X18**, SAXS-Bio	**X1**, Powder Diff.	**X6**, XMCD-Soft
O	**X19**, EXAFS	**X4**, μ-flourescence Imaging	**X11**, GISAXS	**X9**, Magnetic Scat.

2-day experiment

2011 Neutron/X-ray Scattering Experiments and Descriptions

Neutron Experiments

N1. Triple-Axis Spectrometers (HB-1A, HB-1, HB-3)

"Spin wave and phonon dispersion in Fe-Ga solid solutions"
Jerel Zarestky, Masaaki Matsuda, Mark Lumsden

Fe-Ga alloys with appropriate composition and heat treatment, exhibit giant magnetostriction in a polycrystalline and ductile form. The tetragonal magnetostriction coefficient, λ100, of Fe-Ga can be up to 15 times that of pure Fe. This makes these materials of tremendous scientific and technological interest for use in devices such as actuators, transducers and sensors. Elastic constant measurements show that the shear elastic constant $1/2(C_{11}-C_{12})$ decreases with increasing gallium concentration and extrapolates to zero at approximately 26 at.% Ga. The slope of the phonon dispersion curve at low-q of the T_2[110] branch is a measure of that elastic constant and hence the interest in measuring phonons in these materials. With the large magnetoelastic interactions in such a material, it is also of interest to measure the spin wave dispersion. The triple-axis spectrometers HB-1, HB-1A and HB-3 will be used to measure both phonon and spin waves of three compositions of Fe-Ga.

N2. Neutron Powder Diffractometer (HB-2A)

"Magnetic structure of NiO"
Vasile Garlea, Clarina dela Cruz

Neutron diffraction measurements will be performed to investigate the onset of long-range magnetic order in NiO. Data will be collected at various temperatures, ranging from 600K to 288K, using the Neutron Powder Diffractometer at the HFIR. Rietveld analysis of the crystal and low-temperature magnetic structure will be carried out using FullProf Suite software. The results obtained will be discussed and compared with those reported in earlier studies.

N3. Powder Diffractometer – POWGEN (BL-11A)

"*Powder Neutron Diffraction for crystal structure refinement and quantitative phase analysis"*
Ashfia Huq, Jason Hodges, Olivier Gourdon, Luke Heroux

The student groups will have the opportunity to fill a sample holder with sample powder and perform a helium gas pump-purge of the holder, readying it for neutron diffraction with our FERNS sample changer. They will learn how to set up a run using the Data Acquisition System (DAS) and also reduce data using MantidPlot generating GSAS & Fullprof normalized diffraction data files. Afterwards they will learn Rietveld refinement using Powgen time-of-flight (TOF) neutron diffraction data. Exercises will include

- Sample 1: A simple structure (Ni or LaB6) to introduce TOF refinement concept.
- Sample 2: Quantitative phase analysis (NIST standard 674b: a mixture of ZnO, TiO2, Cr2O3 and CeO2).
- Sample 3: Finally for those who want to refine a more complex structure, we will look at several models to determine the true crystal structure of Ba2CuWO6 which shows a Jahn-Teller distortion. This example will also be discussed earlier in the lecture.

Students who do this experiment will have the option of bringing one of their own samples for which data will be collected and analyzed at the school. Samples should be 2-3 cubic cm of powdered sample. The sample can be a loose powder or pressed into pellets or cylinders, and must fit into a 1 cm diameter x 5 cm can. Data can be collected at a temperature between room temperature and 15K.

2011 Neutron/X-ray Scattering Experiments and Descriptions

N4. Four-Circle Diffractometer (HB-3A)

"Structure and lithium-ion motion in the triphylite LiFePO4 studied by single crystal diffraction"
Huibo Cao, Bryan Chakoumakos

Triphylite (LiFePO4) is a promising cathode material for lithium ion batteries due to its virtues of low cost, better safety characteristics, environmental friendliness. But it also faces a significant challenge to achieve both high reversible lithium storage capacity and rapid ion and electron transport capabilities for large-scale EV applications. Studies on the lithium-ion motion properties will help to understand the lithium conduction mechanisms in a lithium ion battery. Using single crystal neutron diffraction, we will resolve the structure of a natural triphylite single crystal at several selected temperatures. Besides the nuclear structure, we are also able to give the magnetic structure at the temperatures lower than its transition temperature. Fullprof and Shelx will be used to refine both nuclear and magnetic structures.

N5. General-Purpose Small Angle Neutron Scattering – SANS (CG-2)

"*Micellar Morphologies in Self-Associated Triblock Copolymer Solutions"*
Ken Littrell, Yuri Melnichenko

The PEO-PPO-PEO triblock copolymers have important applications in industry and medicine. Because of the differing solubilities of PEO and PPO in water, these copolymers exhibit a rich phase behavior that is sensitive to polymer concentration, solvent ionic strength, temperature, and pressure. These phase changes occur by the self-assembly of the polymer chains into structures with characteristic length scales most appropriately measured in nanometers. Thus, small-angle neutron scattering (SANS) is a probe uniquely well-suited to studying this phase behavior. In these experiments we will probe the effects of concentration and ionic strength on block copolymer self-assembly using solutions of 1,2, and 5 wt% Pluronics F108 triblock copolymer in D20 with varying concentrations of salt added, one series in which the anion is the same and the cation is varied, and another where the reverse is true. The size, morphology, and aggregation number of the micellar structures will be extracted through nonlinear least-squares fitting of the scattering data to model functions.

N6. Biological Small Angle Neutron Scattering – Bio-SANS (CG-3)

"Protein unfolding studied by small-angle neutron scattering"
Volker Urban, William Heller

Small-angle neutron scattering (SANS) is a powerful tool for looking at the conformation of biological macromolecules in solution. SANS is particularly sensitive to conformational changes of proteins and nucleic acids in response to applied stimuli, such as temperature, pressure or small molecules. We will study the solution conformation of human serum albumin, a multifunction protein found in the blood, and how it changes in response to urea, a protein denaturant, using the Bio-SANS instrument at HFIR. Various methods of fitting the data will be employed to extract the molecular weight of the scattering particle, the radius of gyration, the distance distribution function P(r) and the maximum linear dimension. Methods for developing models of the protein from SANS data will also be discussed.

2011 Neutron/X-ray Scattering Experiments and Descriptions

N7. Extended Q-range Small Angle Scattering Diffractometer – EQ-SANS (BL-6)

"Conformational Studies of Dendrimers by Small Angle Neutron Scattering"
JK Zhao, Dazhi Liu, Carrie Gao

Dendrimers are a class of regularly branched macromolecules synthesized from a multifunctional core with a succession of iterative processes that double the number of reactive termini for each generation. Globular in shape like spherical colloids, they exhibit additional internal degrees of freedom which give rise to rich structural and dynamic features. Dendrimers possess an array of highly desirable properties that makes them excellent candidates for templates for specific drug and gene delivery agents for cancer therapy. In this experiment, students will learn the basic concepts and skills of SANS by studying the conformational properties of dendrimers. Students will learn how to load samples and acquire data at the Extended Q-range Small Angle Neutron Scattering (EQ-SANS) spectroscopy. Students will also practice how to reduce the data and fit the data with a fuzzy shell spherical model. The conformational parameters will be extracted from the model fitting.

N8. Quasi-Elastic Neutron Scattering – BASIS (BL-2)

"Diffusion dynamics of protons in a novel ionic liquid designed for proton-exchange membranes"
Eugene Mamontov, Souleymane Omar Diallo, Niina Jalarvo, Suresh Mavila Chathoth, Xiang-Qiang Chu

Protic ionic liquids show great potential for mobile fuel cell applications. They possess appealing features such as almost negligible vapor pressure, the characteristic electrical conductivity of an ionic conductor, and a sizable temperature gap between the melting and decomposition points. The diffusion dynamics of protons in these complex liquids are closely tied to their performance as electrolytes. Quasielastic neutron scattering (QENS) is a technique of choice for studying the details of diffusion dynamics of hydrogen because of (1) the large incoherent scattering cross-section of hydrogen compared to other elements and (2) capability of probing spatial characteristics of diffusion processes through dependence of the scattering signal on the momentum transfer, Q. The latter is a clear advantage of QENS compared to, for instance, NMR. In our QENS experiment to be performed on the new SNS backscattering spectrometer, BASIS, we will utilize the Q-dependence of the scattering signal to identify and analyze several dynamic processes involving diffusion motions of hydrogen atoms in a recently synthesized ionic liquid [H2NC(dma)2][BETI].

N9. Fine-Resolution Fermi Chopper Spectrometer – SEQUOIA (BL-17)

"Dynamics of metal hydride systems: Harmonic oscillators and beyond"
Garrett Granroth, Sasha Kolesnikov, Todd Sherline

The hydrogen in zirconium hydride (ZrH2) sits at the interstitial positions between the zirconium. At the simplest description, the energy levels can be considered to be the same as a particle in a potential well. The aim of this experiment is to measure the vibrational spectrum of ZrH2 as a function of energy and wave vector transfer and determine how well it conforms to the predictions of the scattering from a harmonic oscillator. Practical applications of sample preparation, data collection and analysis will be given to generate the scattering function $S(Q,\omega)$ from the data. This will be compared to theoretical predictions based on the harmonic oscillator description, with a discussion of what may cause any discrepancies found. As time permits, other metal hydrides will be measured to highlight differences in their energy spectra.

2011 Neutron/X-ray Scattering Experiments and Descriptions

N10. Spallation Neutrons and Pressure Diffractometer – SNAP (BL-3)

"Pressure-induced phase transitions of water at room temperature"
Antonio dos Santos, Jamie Molaison, Neelam Pradhan, Georgios Karotsis

Students will load a sample of liquid water into a Paris-Edinburgh pressure cell. They will increase the pressure on the sample first to 1.5 GPa and then to 3 GPa, collecting data at each point. Once analyzed, the data will reveal that the sample has undergone two phase transitions: first from liquid water at ambient pressure to ice VI at 1.5 GPa and second from ice VI to ice VII at 3 GPa.

N11. Engineering Materials Diffractometer – VULCAN (BL-7)

"In-situ neutron diffraction measurement of intergranular strain evolution in 316 stainless steel under uniaxial loading at VULCAN"
Ke An, Ducu Stoica, Harley Skorpenske

Anisotropic materials such as stainless steels will develop strong intergranular strains in the regime of plastic deformation. Neutron diffraction allows strain/stress measurement at depth by its high penetration through most engineering materials. The lattice strains of different lattice plane can be calculated by Bragg peak shift with respect to zero strain/stress a reference. At spallation neutron source, using time-of-flight materials science and engineering diffractometer VULCAN can probe changes of lattice strain of all possible *hkl* directions under in-situ loading. In this experiment, a cubic fcc stainless steel dog bone sample of 6 mm in diameter will be applied tensile loading continuously up to 5% engineering strain by using the VULCAN MTS loadframe. In the meantime neutron diffraction pattern of the steel sample will be collected. The neutron data will be separated and reduced based on the load intervals. Single peak refinement will be used for analyzing the intergranular strains of [111], [200], [220] and [311] lattice plane in the material under uniaxial loading. Through this practice, students will learn in-situ loading neutron diffraction measurement set-up at materials science and engineering diffractometer VULCAN, lattice strain data calculation from diffraction pattern using VDRIVE software, and understand the nature of intergranular strain evolution of material under loading.

N12. Magnetism Reflectometer (BL-4A)

*"Revealing magnetism in thin films of normally **non-magnetic** materials"*
Valeria Lauter, Haile Ambaye, Richard Goyette

Understanding the magnetic properties of complex materials near surfaces and interfaces is of critical importance for the development of functional nanostructures and devices. To investigate such structures, where the magnetic layer is only a few unit cells thick and buried within a material, polarized neutron reflectometry is clearly the method-of-choice. During the last two decades Polarized Neutron Reflectometry (PNR) has become a very powerful and popular technique in the study of properties of thin films and multilayers. Recent studies show a strong influence of interfaces on the magnetic properties of thin films, leading to behaviors that are radically different from those of bulk materials. Students will apply polarized neutron reflectometry to the study of interfacial magnetism in $LaMnO_3$-thin film eptaxially grown on $SrTiO_3$ substrate. They will mount the sample in the Displex and will learn how to align the sample in the neutron beam of only 50 microns thick. First PNR measurement will be performed at room T. Then the sample will be cooled down to 5K and the measurement will be repeated. The students will process the data using the data reduction programs and will compare the results of the two experiments. With this practice, students will learn polarized neutron reflectometry set-up, in-situ data reduction from 2-dimensional intensity maps, and understand the evolution of properties in thin films with temperature.

2011 Neutron/X-ray Scattering Experiments and Descriptions

N13. Liquids Reflectometer – horizontal surface (BL-4B)

"Polymer self-diffusion studied by specular reflectivity"
John Ankner, Jim Browning, Candice Halbert

Isotopic substitution is a powerful tool in neutron scattering studies. In this experiment we will observe the self-diffusion of polystyrene (PS) by means of a 500-Å-thick deuterated (dPS) layer float-deposited atop a spin-coated 500-Å-thick protonated PS layer on a silicon substrate. Students will prepare the film in the beamline 4B wet lab and measure specular reflectivity. We will then anneal the sample for ~30 mins in a vacuum oven and re-measure the reflectivity. Students will fit the data from the two runs to observe changes in the interfacial width of the dPS/PS.

2011 Neutron/X-ray Scattering Experiments and Descriptions

X-ray Experiments

X1: Powder diffraction – Area Detector

"In situ x-ray powder diffraction and analysis"
Greg Halder and John Okasinski

Students will participate in making *in-situ* x-ray powder diffraction measurements using a two-dimensional area detector that enables rapid data collection to investigate crystallographic changes during materials processing. Practical aspects relating to sample preparation, experimental set-up, and optimizing collection variables for *in-situ* measurements will be demonstrated. Data from a sample will be collected during a thermal profile program. Sequential GSAS (or FullProf) Rietveld analysis will then be used to determine changes to the lattice parameter.

X2: High Energy Diffraction – Stress & Strain, 1-ID

"Strain and texture measurements in polycrystalline bulk samples using high-energy X-rays"
Jon Almer

Polycrystalline materials encompass large groups of materials such as metals, ceramics and minerals. For many applications it is crucial to understand the structure-performance relationships of such materials under thermo-mechanical processing, e.g. rolling, annealing. We concentrate here on the internal strains and stresses as well as grain orientation aspects (texture). The properties often depend on the local position within the sample and may be mapped if the spatial resolution of the probe is sufficient. The dynamical behavior at surfaces is often not representative of the bulk due to effects such as stress relaxation or abnormal grain growth. Therefore a bulk penetrating probe is required such as high energy X-rays (40 to 100 keV). Third generation high-energy synchrotrons like the APS provide high energy X-rays of unprecedented brilliance enabling high spatial resolution and, in combination with 2D-detectors, fast data acquisition. High energy X-rays are therefore particularly suited for in-situ investigations and rather complementary to neutrons, which in general provide even higher penetration power but substantially coarser spatial resolution and slower data acquisition. In this experiment we will use high-energy x-rays to monitor strain and texture in a polycrystalline sample under in-situ mechanical loading.

X3 & X15: X-ray Tomography, 2-BM or 13-BM

"X-ray computed microtomography (CAT scans) of porous media and corrosion in Al"
Mark Rivers (13-BM) or Francesco Decarlo and Xanghui Xiao (2-BM)

X-ray computed microtomography will be performed on beamlines 2-BM or 13-BM. How x-ray tomography is performed including basic tomography principles, sample mounting and alignment, data collection, data analysis and 3D rendering will be covered. On 13-BM samples containing solids, water, and oil will be measured. The water will be doped with cesium and the oil doped with iodine. By collecting tomographic data sets above and below the K x-ray absorption edges of these elements the complete 3-D distribution of the liquids and solids will be determined. Such information can be used, for example, to study the efficiency of environmental remediation techniques, or of methods for enhanced petroleum extraction. On 2-BM tomographic data will be used to look at corrosion in Aluminum structures.

2011 Neutron/X-ray Scattering Experiments and Descriptions

X4: X-ray micro-fluorescence imaging of bio-samples, 2-ID-E

"Trace element micro-analysis of biological cells by X-ray fluorescence microscopy"
Sophie-Charlotte Gleber, Jesse Ward, Stefan Vogt, and Lydia Finney

Metals and other trace elements are essential for the existence of life as we know it. In any organism, there are only few intracellular processes that do not depend on the presence of metals or other trace elements. Hard x-ray fluorescence microscopy is a powerful technique to study the distribution and chemical state of the elements from Al, P to Cu, Zn and above, with high spatial resolution and very high sensitivity. Due to its inherent low background, x-ray fluorescence is particularly well suited to detect elements present only in trace quantities, down to the level of attograms. The elemental content is measured directly by using the characteristic fluorescence of atoms excited by the microfocused X-ray beam, without the need for element-sensitive dyes. In this experiment, we will map and quantify the elemental distributions of elements from Si to Zn in single cells, in mouse tissue sections, and correlate these with visible light micrographs obtained from the same samples.

X5: Nuclear Resonant and Inelastic X-ray Scattering 3-ID & 30-ID

"Quantitative probing of iron elasticity with high energy resolution X-ray scattering"
Ahmet Alatas, Ayman Said, Bogdan Leu, and Ercan Alp

Elastic properties can be probed quantitatively with two high energy resolution techniques (inelastic X-ray scattering IXS and nuclear resonant inelastic X-ray scattering NRIXS), as it was recently shown in biological and geophysical studies. IXS probes collective excitations, yielding sound velocities and dispersion relations. NRIXS is a highly site-selective technique, which produces the partial density of states of the nuclear resonant isotope (57Fe in this case). We will run two separate experiments on an iron foil to measure the longitudinal (compressional) sound velocity (IXS, sector 30) and the Debye sound velocity (NRIXS, sector 3). From these quantities and the density of the material we will calculate a series of elastic parameters, including the bulk modulus.

X6 & X7: X-ray magnetic circular dichroism - 4-ID-C or 4-ID-D

"Element selective magnetization measurements using XMCD"
John Freeland, Yong Choi, and Jonathan Lang

X-ray magnetic circular dichroism (XMCD) measures the difference in absorption of circularly polarized x-rays by a magnetic material. This technique can be used to extract element and orbital specific magnetic information. In this experiment spectra will be taken at either the soft (C) or hard (D) x-ray beamlines on APS-4-ID. Most of the absorption edges that probe the primary magnetic electrons (3d and 4f) lie in the soft x-ray portion of the spectrum, which requires a windowless UHV (soft x-ray) beamline. Using soft x-rays, XMCD spectra will be taken of a trilayer film. The XMCD spectra as a function of applied magnet field will be taken for different elements to determine the field required to switch individual layers in the material. Using hard x-rays (~8000 eV), XMCD spectra will be taken of a rare-earth/transition-metal compound at several temperatures to determine the compensation temperature in the material.

2011 Neutron/X-ray Scattering Experiments and Descriptions

X8, X14, & X18: Small Angle X-ray Scattering , 5-ID, 12-ID, or 18-ID

"Small-Angle X-ray Scattering from Dilute Monodispersed Proteins or RNA"
Steven Weigand (DND-CAT) or Liang Guo (Bio-CAT)

Students will do SAXS measurements on either of two beamlines. On beamline 5-ID (DND-CAT), small-angle X-ray scattering data will be collected and analyzed from several dilute protein solutions using the in-vacuum capillary flow-cell. Students will also get experience with the ATSAS software package, as they construct dummy-atom models based on the data collected. On 19-ID (Bio-CAT), students will have chance to collect small-angle x-ray scattering data of protein or RNA samples in solution, reduce data from 2D to 1D format and perform basic analysis on the reduced data. Students will also gain experience in using available modeling programs to construct low-resolution models of proteins and RNAs.

X9: Magnetic X-ray Scattering, 6-ID

"Resonant magnetic x-ray scattering from TbNi2Ge2y single crystal"
Zahir Islam and Jong-Woo Kim

This experiment will go over the basics of aligning a single crystal in a diffractometor. Magnetic Bragg diffraction peaks from a TbNi2Ge2 single crystal will be measured and their intensity compared to that of the structural charge peaks on and off resonance. The size of the moment and wave vector of the magnetic peak will be measured as a function of temperature.

X10: Time-resolved x-ray diffraction, 7-ID

"Time-resolved x-ray diffraction from semiconducting materials"
Don Walko and Eric Dufresne

This experiment will consist of laser-pump/x-ray diffraction-probe measurements of crystalline solids. An ultrafast Ti:sapphire laser will be used to excite a variety of materials systems. X-ray Bragg diffraction is used to probe the response of crystalline matter to the laser, with a time resolution limited by the length of APS x-ray bunches (~100 ps). The laser is synchronized to the APS accelerator, with electronics that can vary the delay time between the arrival of the laser and the x-rays at the sample. The first part of this experiment will be to use the laser to excite coherent acoustic strain waves in a semiconductor sample; the strain-induced deformation of the Bragg peak will be observed. The second part of this experiment will be to use the laser to heat a thin metal film grown on a transparent substrate. The time-dependent shift of the film Bragg peak will act as a thermometer for the film, from which the conductance of the film/substrate interface will be measured.

X11: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID

"GISAXS from organic photovoltaic thin films"
Wei Chen, Zhang Jiang, Joseph Strzalka

Since their introduction in the mid-90's, organic photovoltaics (OPV) based on the polymer:fullerene bulk heterojunction (BHJ) have become a fast-growing area of research, resulting in steady improvement in solar cell efficiencies from approximately 1% to over 8 %, approaching the 10% efficiency expected to result in their widespread commercialization . This inexpensive and scalable technology promises to play an important role in meeting the world's energy needs. Understanding and further optimizing OPV technology requires, in part, insights into how the morphology of these thin film devices affects their function, and how different processing conditions influence the morphology and hence the solar cell efficiency. Grazing

incidence x-ray scattering (GIXS), which can non-destructively probe statistically meaningful regions and reveal hierarchical structure on lengthscales varying from Angstroms to tens or hundreds of nanometers on surfaces or buried interfaces, has become an essential tool for this effort. Participants will measure and analyze GIXS from thin film samples of typical OPV material processed under different conditions, thereby gaining direct experience of the kind of information that can be gained from these measurements, as well as familiarity with the hardware and software in use at 8-ID-E.

X12: High Resolution Powder Diffraction, 11-BM

"Pocket Full of Kryptonite"
Matthew Suchomel, Bryan Toby, Lynn Ribaud, & Bob VonDreel

Students will be given a chance to participate in robotic-assisted data collection on the 11-BM high-resolution 12-analyzer powder diffractometer. They will use customized beamline software to prepare and perform scans on a sample of the mineral Jadarite. Students will be given the opportunity to learn about the Rietveld method, and will use GSAS to perform their own Rietveld refinement of collected 11-BM data. In the end, a three-dimensional atomic structure of the Jadarite sample will be determined; a compound that has the identical chemical formula as marked on the crate of 'kryptonite' Lex Luthor obtained in “Superman Returns”. This 'kryptonite', however, should only help your x-ray vision. Instructional material on GSAS and the Rietveld method will be provided.

X13: Pair Distribution Function, 11-ID-B

"Pair-Distribution-Function measurements with High-Energy X-rays."
Peter Chupas and Karena Chapman

High-energy X-rays will be used to measure the structure function to a high value of momentum transfer, Q. Further normalization of the structure factor and subsequent direct Fourier transformation will yield the Pair-Distribution-Function (PDF). The PDF measures local atom structure by recovering atom-atom correlations on a length-scale up to several nanometers. The strength of the technique is that is does not require assumptions of translational symmetry that traditional crystallographic approaches do and thus PDF has been used to study disordered materials from glasses to nanoparticles. The experiment will cover strategies of data collection and processing, and simple modeling approaches.

X16: Ultra-Small angle X-ray Scattering, 15-ID

"USAXS studies of structure of common materials"
Jan Illavsky, Peter Jemian, and Byron Freelon

The USAXS experiment is World-unique facility for small-angle scattering in unprecedented scatterers size range – from nanometers to microns during one measurement. Engineering materials – metals, polymers, ceramics, etc. – often exhibit complex, hierarchical, microstructures spanning this wide range of sizes. Students will become familiar with this unique technique and measure selected examples of materials they may use during their day-to-day life. Analysis of the USAXS data using general purpose *Irena* software will be showcased as part of the experiment. DVD with software distribution and instructional material will be provided to students.

2011 Neutron/X-ray Scattering Experiments and Descriptions

X17: High-Pressure Powder Diffraction, 16-BM-B

"Pressure-induced structure phase transition in ZnO"
Yue Meng

In this experiment, students will get familiar with the high-pressure XRD experiment procedure, observe the pressure-induced structural phase transition in ZnO using angle dispersive x-ray diffraction technique, and refine unit cell parameters of the low- and high-pressure phases of ZnO at high pressure.

X19. X-ray Absorption Fine Structure - 20BM

"Polarization Dependent XAFS in High Tc Superconductors"
Steve Heald

The polarization dependence of the XAFS can be very powerful in separating contributions from various bonds in layered materials. This will be demonstrated with measurements on cuprate-based High Tc superconductors. Oriented powder samples will be used along with polarized synchrotron radiation to isolate the XAFS signals from in-plane and out-of-plane bonding. This data will be analyzed using simple linear combination fitting for the near edge region, and first shell analysis for the EXAFS.

X20: X-ray nano-diffraction, 26-ID

"Ferromagnetic phase nucleation and growth observed via variable-temperature nanofocused x-ray diffraction microscopy "
Jong-Woo Kim, Philip Ryan, Martin Holt, Volker Rose, Robert Winarski

Epitaxial films of FeRh on MgO substrate is an antiferromagnet (AFM) at room temperature but when heated above the transition temperature (~370 K), it undergoes a first order magnetic transition and becomes a ferromagnet (FM) with a lattice expansion about 0.7 %. Due to this lattice expansion, the two structural phases can be identified by the x-ray diffraction. The clear peak separation at the middle of the transition implies the coexistence of the AFM and FM phases in normal x-ray diffraction. This two-phase coexistence and the relationship of nucleation, growth, and nanoscale critical phenomena to film defects and strain will be observed via variable-temperature diffraction microscopy at the Hard X-ray Nanoprobe (HXN) beamline

X21. Transmission X-ray Microscope, 32-ID

Steve Wang

Lens-based x-ray microscopy and nano-CT is a relatively new approach for imaging and characterizing nano-structures. To date, tens of nanometer resolution in 2D and 3D is routinely achieved with both commercial laboratory systems and instruments based at synchrotron radiation facilities. The non-destructive nature of the technique makes it particularly well suited for *in situ* studies on dynamic behavior of nanostructures while in or near their real operation conditions. With the use of tunable synchrotron x-ray sources, the elemental and certain chemical compositions can also be mapped accurately in 3D with 1-percent level sensitivity. We will provide an introduction to this technique with examples in several application areas.

2011 Neutron/X-ray Scattering Experiments and Descriptions

X22: Grazing incidence interface diffraction, 33-BM

"Grazing incidence x-ray diffraction study of atomic modulations in ordered oxide films"
Phil Ryan and Jenia Karapetrova

The synthesis of complex oxide superlattices with single unit cell control and atomically sharp interfaces has opened new routes to stabilizing collective ordering phenomena in materials. Heterostructures of dissimilar complex oxides have received considerable interest due to the novel interfacial properties that emerge resulting from the competition between the spin, charge, or orbital ground states of the adjoining compounds. Superlattices can exhibit magnetic ordering temperatures that much higher than those measured in compositionally equivalent alloys. This experiment will use grazing incidence x-ray scattering to measure the structural properties of the superlattice and how it is related to the magnetic order.