Neutron Experiment descriptions:

N1: Triple-Axis Spectrometers, HFIR HB1A & HB3

Spin wave and phonon dispersion in Fe-Ga solid solutions

Fe-Ga alloys with appropriate composition and heat treatment, exhibit giant magnetostriction in a polycrystalline and ductile form. The tetragonal magnetostriction coefficient, λ_{100}, 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-1A and HB-3 will be used to measure both phonon and spin waves of two compositions of Fe-Ga alloys.

N2: Powder Diffractometer, HFIR HB2A

Magnetic structure of NiO

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: Four-Circle Diffractometer, HFIR HB3A

Structure and lithium-ion motion in the triphylite LiFePO4 studied by single crystal diffraction

Triphylite, $Li(Fe,Mn)PO_4$, is a promising cathode material for lithium ion batteries due to its virtues of low cost, better safety characteristics and 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.

N4: Neutron Imaging Station, HFIR CG1D

Dynamics of fluid flow in permeable rock

The principle of neutron imaging is based on the attenuation from both absorption and scattering, of a directional neutron beam by the matter through which it passes. Neutron imaging is complementary to other imaging techniques such as X-rays. X-rays are scattered and absorbed by electrons, so absorption and scattering increase monotonically with atomic number. Neutrons, on the other hand, interact with nuclei and their scattering power does not vary in any regular way with atomic number. Several areas of research already benefit from neutron imaging, such as engineering, advanced material characterization, fluid-flow and/or two-phase flow devices, automotive technology, advanced manufacturing technology, applied sciences, aerospace, life and biological sciences, national security applications, etc. Neutrons are specifically well suited for imaging light atoms (hydrocarbons for example) buried in heavy atoms, and are capable of characterizing fluid flow (dynamics). Time resolved water uptake and flow in permeable rocks will be spatially mapped and measured via neutron imaging.

N5: Small Angle Neutron Scattering, HFIR CG2 General Purpose SANS
HFIR CG3 Bio-SANS
SNS BL6 EQ-SANS

Micellar morphologies in self-associated triblock copolymer solutions: effects of concentration and contrast matching in porasils

The PEO-PPO-PEO triblock copolymers have important applications in industry and medicine. Because of the different 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 of the order of few nanometers. Thus, small-angle neutron scattering (SANS) is a technique uniquely well-suited to studying this phase behavior. In these experiments we will study 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 D_2O 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.

Contrast-matching SANS has been widely used to characterize structure of soft and biological matter as well as pore accessibility in porous materials. The particular advantage of this technique is attributed to the large difference in coherent scattering lengths of hydrogen and deuterium. By changing composition of protonated and deuterated solvent (such as H_2O and D_2O), one can vary the average scattering length density of the solvent and hence vary the contrast between the scattering objects and surrounding medium. In this experiment, three porasil samples (porous silica) with different H_2O/D_2O ratios (empty pores, i.e. full neutron contrast), pores filled with 71% H_2O + 29% D_2O (intermediate neutron contrast) and 42%H_2O + 58%D_2O (zero-average contrast)) will be measured to demonstrate the power of contrast matching SANS technique.

N6: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS BL1B

Introduction to Pair Distribution Function analysis

The Nanoscale Ordered Materials Diffractometer (NOMAD) is designed for the determination of pair distribution functions (PDF). The PDF is a measure of the probability to find an atom B at a distance r away from arbitrarily chosen central atom A relative to a random arrangement. As such it is a measure of the atomic arrangement of the sample independent of periodicity and therefore the PDF formalism can be applied equally to liquids, glasses, nanomaterials and long range ordered crystalline materials.

We will determine the PDF of glassy SiO_2 and fit a Continuous Random Network model to it. We will perform an isotope substitution experiment for liquid water. We will look at the PDF of diamond and compare the PDF of bulk and nanocrystalline NiO.

N7: BASIS Backscattering, SNS BL2

Diffusion dynamics of protons in a novel ionic liquid designed for proton-exchange membranes

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 $[H_2NC(dma)_2][BETI]$.

N8: Inelastic Neutron Spectroscopy - INS (VISION), SNS BL16B

High-resolution vibrational spectroscopy with neutrons

The spectroscopic technique implemented at the VISION beam line will be discussed and related to other neutron scattering methods and to Raman- and IR- spectroscopy, the experimental procedures at VISION will be introduced.

We will prepare two samples for use at VISION - Zirconium hydride (ZrH_2) and Toluene. Vibrational data will be collected at low temperature (5K). The raw data will be reduced and normalized with respect to the incident beam spectrum with python based script running in the Mantid framework. The resulting energy transfer spectra will be compared with Raman and/or IR data and data from BL18 (ARCS) if time permits. The spectra will also be compared to theoretical spectra obtained with CASTEP (first-principles quantum mechanical calculations based on plane-wave basis sets and pseudopotential). The expected neutron data can be predicted based on CASTEP results using the a-Climax software.

N9: Magnetism Reflectometer, SNS BL4A

Revealing magnetism in thin films of normally non-magnetic materials

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 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 epitaxially 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-D intensity maps, and understand the evolution of properties in thin films with temperature.

N10: Liquids Reflectometer, SNS BL4B

Polymer self-diffusion studied by specular reflectivity

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 min 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.

N11: VULCAN Engineering Materials Diffractometer, SNS BL7

Non-destructive residual stress/strain measurement of weld by neutron diffraction

Residual stresses in engineering component are important to structure lifetime reliability and durability. During welding, severe residual stresses are commonly built up across the weld metal (WM), heat affected zone (HZ) and base metal (BM). The variation of chemical composition and microstructure will also affect the accurate measurement residual stress. Using Time-of-Flight neutron diffraction on VULCAN instrument, the residual stress/strain and the phase distribution can be spatially resolved by engineering diffraction. In the experiment, a weld-bead-on-plate sample will be used for demonstrating the non-destructive residual stress measurement. A stress-free coupon sample, which has similar chemistry of the weld sample, will be used as the reference. Single peak and Rietveld refinement will be used to determine the residual strain and phase concentration of each measurement location, respectively.

N12: POWGEN Powder Diffractometer, SNS BL11A

Powder Neutron Diffraction for crystal structure refinement and quantitative phase analysis

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 POWGEN Automatic Changer (PAC) 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 LaB_6) to introduce TOF refinement concept.
- Sample 2: Quantitative phase analysis (NIST standard 674b: a mixture of ZnO, TiO_2, Cr_2O_3 and CeO_2).
- 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 Ba_2CuWO_6, which shows a Jahn-Teller distortion.

N13: Wide Angular-Range Chopper Spectrometer (ARCS), SNS BL18

Dynamics of metal hydride systems: Harmonic oscillators and beyond

The hydrogen in zirconium hydride (ZrH_2) 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 ZrH_2 as a function of energy and wavevector 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.

N14: Hybrid Spectrometer (HYSPEC), SNS BL14A

Spin-wave excitations in the orbital-spin coupled system MnV_2O_4

The Hybrid Spectrometer (HYSPEC) is a unique instrument whose concept combines the time-of-flight spectroscopy with the focusing Bragg optics by using the TOF for selecting the neutron energy and a vertically-curved crystal monochromator for concentrating the neutron flux on sample. It is optimized for detailed investigations of the low-energy atomic-scale dynamical properties of crystalline solids. Transition-metal spinels, AB_2X_4, have been for many years the subject of intense experimental and theoretical activity. Structurally, the most interesting feature of these systems is the fact that the B cation occupies the nodes of a pyrochlore lattice, which is known to be geometrically frustrated. In the spinel MnV_2O_4, the octahedral site is occupied by the V^{3+} ion having two 3d electrons in threefold t_{2g} levels. This compound exhibits two transitions to long-range ordered ferromagnetic states, the first collinear and the second noncollinear. The lower temperature magnetic transition is accompanied by a structural distortion to an orbitally ordered tetragonal phase. The HYSPEC spectrometer will be used to investigate the low-energy spin-wave excitations in a MnV_2O_4 single crystal. The exercise will enable students to get hands-on experience with crystal alignment procedures, as well as on data processing using data reduction and plotting programs.

N15: TOPAZ Single-crystal Diffractometer, SNS BL12

High-resolution single crystal structure analysis from 3-D mapping of reciprocal space using TOF Laue diffraction

We will practice the experimental setup, data collection, data reduction procedures and perform a structure refinement of a high-resolution single crystal data set of scolecite measured on TOPAZ using neutron wavelength-resolved TOF Laue technique. Scolecite is the calcium member of the natrolite family within the zeolite group. The cation interaction with the framework oxygen bonding plays an important role in fine tuning the adsorption and electrostatic properties of the porous zeolite channels, which is fundamental for applications in separation science and energy storage materials. Single crystal data collection strategy will be optimized with the locally developed CrystalPlan program; peak integration will be performed in 3D Q-space (reciprocal space) in Mantid. Data reduction including neutron TOF spectrum, detector efficiency, and absorption corrections will be carried out with the ANVRED2X program. The structure will be refined using GSAS. The option to refine the neutron structure in SHELXL97 will also be explored.

X-ray Experiment Descriptions:

X1: High Energy Strain Scattering, 1-ID

"Texture and strain measurement in polycrystalline materials using high energy x-rays"

Jun-Sang Park and Jonathan Almer

Polycrystalline materials encompass large groups of materials such as metals, ceramics, and minerals are employed in wide range of applications. To predict the performance of these materials, it is important to understand the structure–processing–properties relationship. High energy x-ray combined with fast area detectors is an attractive non-destructive tool to investigate this relationship in a polycrystalline material. In this experiment, we will use high energy x-rays to measure the lattice strains and texture in a polycrystalline sample under in-situ mechanical loading.

X2: X-ray Tomography, 2-BM

"X-ray computed microtomography (CAT scans) of porous media and corrosion in Al"

Carmen Soriano Hoyuelos, Francesco Decarlo, Xanghui Xiao

Aluminum alloys are susceptible to intergranular corrosion in wet environments. This is a problem since the narrow regions of attack along grain boundaries can be sites where cracks initiate, leading to structural failure. X-ray microtomography at a synchrotron facility allows to monitor the development of intergranular corrosion in situ in an aqueous environment in real time offering a remarkable insight into the evolution of corrosion enabling us to measure the rate of attack along individual grain boundaries and the rate of growth of the width of the cavities compared with the length, and assess the interaction with microstructural features such as intermetallic particles. In this experiment we will show how x-ray tomography is performed including basic tomography principles, sample mounting and alignment, data collection, data analysis and 3D rendering. Tomographic data will be collected to look at corrosion in aluminum structures.

X3: X-ray Micro-fluorescence Imaging, 2-ID-E & 8-BM

"Trace element micro-analysis of biological cells by X-ray fluorescence microscopy"

Sophie-Charlotte Gleber, Lydia Finney, Stefan Vogt

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 atto-grams. 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.

X4: Nuclear Resonance Scattering, 3-ID

"Nuclear resonant inelastic x-ray scattering"

Michael Hu and Ercan Alp

Nuclear resonant inelastic x-ray scattering (NRIXS) is a synchrotron-radiation-based method that finds a wide range of applications in condensed matter physics, material science, high-pressure research, geosciences, and biophysics. The interaction between nuclei and phonons (atomic vibrations) allow us to study the atomic vibrational properties by probing the nuclei. In an NRIXS experiment, one measures the number of nuclear resonant absorption events as a function of energy transfer from an incident x-ray beam to the sample under study. Vastly disparate energy scales involved in nuclear excitations (many keV) and atomic lattice excitations (tens of meV) implicate the decoupling of these two processes. NRIXS can be described as nuclear resonant excitation plus phonon annihilation or creation. As a result, on the scale of the energies of phonons, the energy of nuclear resonant absorption is modified only through atomic motions in a sample. The unique aspect of using resonant isotopes to measure phonon energies is mainly the selectivity. This means that vibrations can be probed locally in systems that have resonant isotopes in specific places, e.g., bio-molecules like myoglobin, thin films, and materials under extremely high pressure.

X5: X-ray Magnetic Circular Dichroism - 4-ID-C or 4-ID-D

"Element selective magnetization measurements using XMCD"

Yong Choi, David Keavney and Daniel Haskel

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 tri-layer 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.

X6: Magnetic X-ray Scattering, 6-ID

"Resonant magnetic x-ray scattering from a rare-earth compound"

Zahir Islam and Jong-Woo Kim

This experiment will review the fundamentals of aligning a single crystal in a diffractometer. Magnetic Bragg diffraction peaks from a single crystal of a rare-earth compound will be measured and their intensity compared to that of the structural charge peaks. The order parameter and propagation vector of the magnetic peak will be measured as a function of temperature.

X7: Radiography, 7-BM

"Time resolved radiography of liquid fuel sprays"

Alan Kastergren

This experiment will demonstrate the use of x-ray radiography and fluorescence to study both low- and high-speed turbulent flows.

X8: Time-Resolved X-ray Diffraction, 7-ID

"Time-resolved x-ray diffraction"

Don Walko

This experiment will consist of laser-pump/x-ray diffraction-probe measurements of crystalline solids. An ultrafast Ti:sapphire laser can 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. In this experiment, the laser will be used 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.

X9: Grazing Incidence Small-Angle X-ray Scattering (GISAXS), 8-ID-E

"GISAXS from organic photovoltaic thin films"

Zhang Jiang, Joseph Strzalka, Wei Chen

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%, 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 Ångstroms to 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.

X10: X-ray photon correlation spectroscopy, 8-ID-I

"X-ray photon correlation spectroscopy study of dynamics in colloidal suspensions"

Alec Sandy and Suresh Narayanan

X-ray photon correlation spectroscopy (XPCS) is a well-established technique to study the equilibrium dynamics in soft and hard matter systems. XPCS has been successfully applied to study dynamics in colloidal suspensions, nanoparticle dispersion in polymers, polymer thin films, etc. XPCS uses the partially coherent nature of the synchrotron beam to probe speckles and its fluctuations in time. By using a 2-D detector such as a CCD, the dynamics over a range of length scales in the range of 100 nm - 10 nm can be probed simultaneously.

In this experiment, a colloidal suspension of silica spheres in the size range of 100 nm dispersed in a viscous solvent like glycerol will be studied. By varying the particle concentration, single particle Brownian diffusion and the effect of particle interactions will be studied.

X11: X-ray Absorption Near Edge Spectroscopy - 9-BM

"XANES analysis of Pt in catalytic converters"

Trudy Bolin, Tianpin Wu, Steve Heald

This experiment will demonstrate an in-situ catalysis experiment, focusing on the L edge of a 5d metal, commonly used in catalytic processes. For a common Pt-based catalyst, typically used in automobile catalytic converters, the students will see the effects on the Pt L-edge XANES while treating a catalyst in a reducing or oxidizing environment. We will also discuss the chemistry behind the changes in the XANES. Students will also be guided through some of the sample preparation techniques, such as using our "six shooter" to load multiple samples at one time into a reactor, and be guided through starting the experiments.

X12: Synchrotron Powder Diffraction, 11/17-BM

"Hands-on high resolution and in-situ powder diffraction measurements & analysis"

Greg Halder, Saul Lapidus, Andrey Yakovenko, Matthew Suchomel

X-ray powder diffraction is a versatile technique that reveals detailed information about the chemical composition and crystallographic structure of materials, and affords great flexibility for in-situ studies of samples under non-ambient conditions. In this experiment, students will gain hands-on experience with all aspects of modern synchrotron powder diffraction experimentation, from sample preparation to data collection and analysis. Students will become familiar with the world-class suite of dedicated powder diffraction instruments offered at the APS, including both high-resolution and two-dimensional area detectors, and a wide range of in-situ sample environments. They will learn how to access and use these tools for their own science. The second day of this experiment will include an interactive tutorial on Rietveld Analysis methods using the software package GSAS to extract crystallographic structural details from powder diffraction data measured on the first day of the experiment. While this experiment is intended for those new to synchrotron-based powder diffraction, there may be time during the tutorial session for students to ask more in depth questions or get help with the analysis of other APS powder diffraction data.

X13: Pair Distribution Function, 11-ID-B

"Pair distribution function measurements with high-energy X-rays."

Olaf Borkiewicz, Kevin Beyer, Peter Chupas, Karena Chapman

Pair distribution function (PDF) analysis measures local atom structure as the distribution of atom-atom distances from Ångstroms up to several nanometers. A strength of the technique is that it does not assume translational symmetry of the structure, as required for traditional crystallographic approaches, and thus PDF can be applied to study disordered, crystalline, amorphous, nanoscale, homogeneous and heterogeneous materials alike. Experimentally, the PDF is derived from a specialized powder diffraction measurement in transmission geometry: High-energy X-rays are 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 yields the Pair-Distribution-Function (PDF). This experiment will cover strategies for data collection and processing, and simple modeling approaches. We will explore how the experimental variables (beam energy, beam/sample size, detector distance, capillary composition) impact the quality and resolution of the resulting data.

X14: Small Angle X-ray Scattering, 12-ID

"Small Angle Scattering (SAXS) of biological, organic and inorganic systems."

Xiaobing Zuo, Byeongdu Lee

SAXS provides valuable structural information such as size, shape and particle interaction, and has been widely used in material sciences and structural biology. In this experiment, a SAXS pinhole apparatus will be introduced to perform measurements on a variety of different samples like proteins, polymers, nano-particles. The data will be analyzed and interpreted.

X15: X-ray Fluorescence Microtomography 13-ID-E

"Imaging the interior metal distribution of seeds"

Matt Newville and Antonio Lanzirotti

Metals like K, Ca, Mn, Fe, and Zn are important nutrients in plants and seeds, playing different biological roles. Determining what factors control the transport and distribution of these metals in seeds can give important clues to understanding plant genetics and diseases. X-ray Fluorescence (XRF) is highly sensitive to low metal concentrations, and an X-ray micro-beam can give XRF spectra with very high spatial resolution for thin, dense samples. However, the penetrating power of X-rays into light material such as seeds means that a micro-XRF spectrum will average over considerable depth, blurring the spatial resolution. In this experiment, we will combine Computed Microtomography and X-ray Fluorescence, using both the imaging and spectroscopic properties of X-rays. A seed will be rotated and translated through a micro-focussed X-ray beam allowing a virtual slice to be made for each elemental distribution within the seed. The experiment will include mounting and centering the sample, processing the X-ray fluorescence spectra and performing tomographic reconstruction.

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

"USAXS/SAXS/WAXS studies of structure of common materials"

Jan Ilavsky, Peter Jemian, and Joshua Hammons

This instrument provides a unique facility for ultra-small-angle scattering studies over an unprecedented range of lengthscales within a single measurement—from less than Ångstrom to few microns. Engineering materials (e.g. 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, such as toothpaste, food fats, cheese etc. Analysis of the USAXS data using general purpose Irena software will be showcased as part of the experiment.

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

"Pressure-induced structure phase transition in ZnO"

Changyong Park and Dmitry Popov

Pressure is a powerful tool to investigate materials' physical properties like hardness, elasticity, and strength. It can be used to adjust the electrical conductance and magnetism, sometimes leading to a discovery of new superconducting materials with help of combined cryogenic cooling. It also can cause reversible or irreversible phase transitions when the range of pressure is extended beyond the stability field, which many times lead to a discovery of new materials. In the solid state, the range of pressure to cause these physical changes typically goes far to GPa level (Giga Pascal, 1 Pa = $1N/m^2$), for which we need to use a special apparatus, Diamond Anvil Cell (or DAC). In this experiment, students will perform high-pressure powder x-ray diffraction with a pre-loaded DAC sample and learn how it helps to study the materials physical property. The pressure-induced volume contraction and eventually the phase transition in ZnO will be demonstrated and an entry level lattice parameter refinement will be exercised to quantitatively describe the observation.

X18: X-ray Absorption Fine Structure - 20-BM

"Polarization Dependent XAFS in High Tc Superconductors"

Steve Heald, Mali Balasubramanian, Chugjun Sun

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.

X19: Fundamentals of beamline operation, 20-BM

"Fundamentals of beamline operation and Cu XAFS"

Steve Heald and Ercan Alp

There are several parameters that need to be optimized for successful experiments. In the case of x-ray spectroscopy, the most important include the energy resolution, harmonic content, and sample quality (thickness and uniformity). We will work through setting up a beamline, and run several "hands on" exercises looking at these parameters and how they affect the final data. Once the beamline is characterized and properly set up, it will be used to measure two types of Cu samples. An oriented high Tc superconductor sample will be used to illustrate the utility of using the x-ray polarization to isolate signals from the in-plane and out-of-plane bonds. Linear combination fitting of both the EXAFS and XANES will be demonstrated by fitting the data for an arbitrarily oriented sample. We will also measure the Cu foil EXAFS and fit it with the FEFF theory to demonstrate theoretical fitting. Analysis will be done using the Demeter software that can be downloaded from http://bruceravel.github.io/demeter/. Prior experience in synchrotron experimentation is desirable.

X20: X-ray Nano-fluorescence, 26-ID

"Nanoprobe X-ray Fluorescence Studies of Metal Impurity Decoration of Dislocations in Large-Area Solar Cells"

Volker Rose, Martin Holt, Robert Winarski, Ian McNulty, Mariana Bertoni

It is accepted throughout the photovoltaic community that the overall performance of entire modules is regulated by inhomogeneously distributed nanoscale defects inside the wafers. Over the years a variety of techniques have been used to map and characterize precipitates, grain boundaries and dislocations. However, in the race to achieve higher and higher resolutions, while studying industry-relevant material, many of these techniques fall short either due to the inherent resolution limitations of the equipment or because the combination of low defect spatial densities and strong heterogeneity, present a challenge to sample preparation and characterization.

In this experiment, we will demonstrate the future of synchrotron-based nanoprobe techniques for identifying defects in large volumes of commercial solar cell materials. We are going to utilize a state-of-the-art x-ray fluorescence nanoprobe beamline to identify the precise nature of performance limiting defects in commercial mc-Si solar cells. X-ray fluorescence with a beam spot size < 60 nm is used to characterize the contamination levels in solar cell materials.

X21: Inelastic X-ray Scattering, 30-ID

"Inelastic X-ray Scattering"

Bogdan Leu, Ayman Said and Ahmet Alatas

High-energy resolution inelastic X-ray scattering (IXS)—a technique available only at third-generation synchrotron sources—measures phonon dispersion curves in single crystals and probes collective excitations in disordered systems. Traditionally, neutron spectroscopies have been used for this purpose and are still widely used today. However, in numerous cases IXS is preferred (or is the only option) given the very small X-ray beam sizes, which allows for measurements on samples tens of microns in size (e.g., in diamond anvil cells), and the absence of kinematic limitations, ideal for probing acoustic excitations in disordered materials with a speed of sound larger than that of the thermal neutrons.

In this experiment we will emphasize the ability of IXS to measure sound velocities and elastic constants. We will measure the dispersion curves in various directions for an aluminum single crystal and extract the elastic constants and other related quantities (bulk modulus, Young modulus, etc.)

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.

X23: X-ray Micro-Laue Diffraction, 34-ID-E

"Measuring crystal microstructures with x-ray micro-beam Laue diffraction"

Ruqing Xu, Wenjun Liu, Jon Tischler

The x-ray micro-beam Laue diffraction at beamline 34-ID provides a unique diffraction probe of material microscructures with highly-focused, polychromatic x-ray beam and 3D spatial resolution. A pair of custom-profiled K-B mirrors provide sub-micron x-ray focal size, the scanning-wire differential aperture provides depth-resolution along x-ray's penetration, and high-speed area detectors allows 3D mapping over relatively large sample volumes. The technique can reveal detailed local structural information of crystalline materials, such as crystallographic orientation, orientation gradients, grain morphology, strain tensor, and lattice structure, with high spatial resolution of less than 500 nm and angular resolution of 0.01°. It is applicable to single crystal, polycrystalline, composite, deformed, and functionally-graded materials. Applications include studies of fundamental deformation processes, basic grain-growth behavior, electromigration, solid-solution precipitation, structural phase transformation, and high-pressure mineral physics, etc.