2017 Neutron Experiment descriptions:

N1: Triple-Axis Spectrometers, HFIR HB-1 & CTAX

Magnetic excitation and anisotropy in multiferroic $BiFeO_3$

Multiferroic materials, in which spontaneous ferroelectric polarization and magnetic order coexist, have been investigated intensively due to their potential industrial applications. Because the Néel temperature T_N~640 K is much higher than room temperature and because of the large spontaneous electronic polarization (P~100 μC/cm2), $BiFeO_3$ has attracted a lot of attention. We will measure the magnetic excitation in $BiFeO_3$ at room temperature. The excitation energy below 11 meV will be measured at CTAX. In combination with higher excitation energy measured at HB-1, the full magnetic dispersion relation will be determined. The low-energy gapped excitations allow the determination of the Dzyaloshinskii-Moriya interaction and single ion anisotropy.

N2: Powder Diffractometer, HFIR HB-2A

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 HB-3A

Structure and lithium-ion motion in the triphylite $LiFePO_4$ studied by single crystal diffraction

Triphylite, $Li(Fe,Mn)PO_4$, is a candidate 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: WAND powder/single-crystal diffractometer, HFIR HB-2C WAND

Diffuse magnetic scattering in Ho_2PdSi_3

When a neutron beam is diffracted by a sample without translation symmetry the resulting diffraction pattern still carries information. For instance scattering form a liquid

will have a maximum in intensity on the average distance between two particles. In solids, regular stacking faults, intercalated atoms or defects can result in diffuse scattering and the analysis of these scattering patterns is important for understanding the real structure. Diffuse scattering in magnetism can occur in systems where the magnetic exchange interactions allow only degenerate ground states, or where the minimization of energy cannot be achieved for all related magnetic moments. Still, the diffuse scattering can be analyzed to understand the underlying exchange interactions. In this experiment we will investigate the diffuse scattering pattern of a geometrically frustrated rare-earth intermetallic Ho_2PdSi_3. From the diffuse scattering pattern, the spin-spin correlation function is deduced using a reverse Monte-Carlo approach. The spin-spin correlation function is then used to determine the ordered magnetic ground state. Results between simulation and magnetic ground state are used to evaluate the advantages and limitations of this technique. The experiment will include the careful hands-on set-up of the sample at the experiment using a neutron camera and goniometer, and the data treatment for the Monte-Carlo simulation.

N5: Neutron Imaging Station, HFIR CG-1D

Neutron imaging of metals exposed to high temperatures

The neutron imaging team is developing high temperature metal casting capabilities at the CG-1D imaging beamline. A wide range of interesting behaviors may be observed during the melting and re-solidification of metal alloys, including the evolution of composition gradients, phase separation, and porosity, as functions of temperature and time. Dynamic casting studies will be conducted on metal alloys containing elements such as Fe, Co, and Cr, which have sufficient neutron attenuation and contrast needed to observe their casting evolution. Another type of in situ investigation is based upon the famous Kirkendall effect, where the sharp interface between different metals broadens and shifts due to the vacancy-assisted diffusion of the different elements. The in situ diffusion and casting experiments address important issues, such as porosity evolution as a function of temperature, in science areas such as additive manufacturing and the development of advanced alloys, such as the high-entropy alloys (HEAs) containing four or more elements.

N6: Small Angle Neutron Scattering, HFIR CG-2 General Purpose SANS HFIR CG-3 Bio-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 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.

N7: Engineering Materials Diffractometer, HFIR NRSF2

Non-destructive residual stress/strain measurement of friction stir welded ODS steel

"Engineering Diffractometers" are neutron diffractometers with fine collimation of the incident and diffracted beams that can be used to obtain diffraction patterns from small well-defined volumes inside bulk materials. The diffraction pattern can be analyzed to identify and quantify the crystalline phases present, the degree of preferred orientation, and deviations from the stress-free lattice parameters (i.e., strain), which indicate residual stress. Residual stresses in engineering components are important to structure lifetime, reliability and durability. Mechanical processing, extrusion, bending, forging, and joining of metals all can result in significant residual stress in engineering components, and these stresses directly impact service life. This project will focus on how engineering diffractometers at both a spallation source (VULCAN) and a reactor source (NSRF2) have unique advantages which can be used to characterize complex materials using the friction stir welding plate as an example.
Friction stir welding (FSW) is a solid-solid joining process designed to avoid many of the drawbacks associated with conventional welding. Even so, significant residual stresses can be generated across the weld metal (WM), thermo-mechanical affected zone (TMAZ) and base metal (BM). Using the NRSF2 diffractometer, we will determine the residual stresses from FSW in an experimental oxide dispersion strengthened (ODS) alloy. Discussion of the proper selection of an unstressed lattice spacing (d_0) will be performed. Single peak fitting of data from NRSF2 will be used to determine the residual strain and phase concentration of each measurement location, respectively. The engineering diffractometer VULCAN at the SNS will also be discussed and contrasted with the NRSF2 instrument, and a sample dataset from VULCAN will be analyzed.

N8: BASIS Backscattering, SNS BL-2

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

N9: VISION Inelastic Neutron Spectroscopy, SNS BL-16B

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.

N10: Magnetism Reflectometer, SNS BL-4A

Revealing magnetism in thin films of normally non-magnetic materials

Understanding the magnetic properties of complex materials near surfaces and interfaces critically important 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 study interfacial magnetism in a

$LaMnO_3$-thin film epitaxially grown on a $SrTiO_3$ substrate. They will mount the sample in the Displex and will learn how to align a sample with a footprint of only 50 microns wide in the neutron beam. First PNR measurement will be performed at room T. Then the sample will be cooled 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.

N11: 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.

N12: NOMAD Nanoscale-Ordered Materials Diffractometer, SNS BL-1B

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 $BaTi_2O_5$. We will introduce real-space fitting using the 'small-box' refinement program PDFgui, modeling the PDF of diamond, crystalline SnO_2, and SnO_2 nanoparticles. We will also introduce the levitation sample environments at NOMAD for container-less and high temperature neutron scattering, performing a laboratory experiment with a melt.

N13: POWGEN Powder Diffractometer, SNS BL-11A

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). 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: 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.
- Sample 4: Finally, those who get through the first three examples will be able to learn how to do sequential refinement for temperature scans of ZrW_2O_8.

N14: SEQUOIA Fine-Resolution Fermi Chopper Spectrometer, SNS BL17

Dynamics of metal hydride systems: Harmonic oscillators and beyond

The hydrogen in zirconium hydride (ZrH2) sits at the interstitial positions between the zirconium. At the simplest description, the energy levels are 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. As time permits, other metal hydrides will be measured to highlight differences in their energy spectra.

N15: TOPAZ Single-crystal Diffractometer, SNS BL-12

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 ($CaAl_2Si_3O_{10}\cdot 3H_2O$) 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 ANVRED3 program. The structure will be refined using GSAS. The option to refine the neutron structure in SHELX 2014 will also be explored.

N16: HYSPEC Hybrid Spectrometer, SNS BL14A

Separating nuclear and magnetic scattering from MnO using neutron polarization analysis

Because neutrons have a magnetic moment, they can scatter from atomic-scale magnetic structures, and can create or destroy quantized excitations that have a magnetic character in materials. By utilizing polarization filters, magnetic guide fields and what we call 'spin flippers', we can preferentially select neutrons of a single orientation, preserve or steer that orientation, and invert the orientation with respect to the guide field. These tools enable us to distinguish between scattering events which preserve or invert neutron polarization, enabling a technique we call XYZ polarization analysis. Here, XYZ refers to the ability to reorient the guide field at the sample position in orthogonal directions using an array of electromagnetic coils. In this experiment, we will demonstrate the use of polarization analysis to separate the nuclear and magnetic scattering from a manganese oxide, MnO, powder sample. This material is considered a benchmark antiferromagnet, and exhibits long-range ordering at temperatures below 118 K. The magnetic moments are arranged in ferromagnetic sheets parallel to (111) planes, and the direction of magnetization in neighboring planes is antiparallel. Polarized neutrons are used to unambiguously identify the magnetic Bragg scattering and the spin-wave excitations from the ordered state, as well as the diffuse scattering that persists well above the ordering temperature. The exercise will enable students to get hands-on experience with the polarized neutrons scattering technique, as well as on data processing and visualization using Mslice and Mantid packages.
This experiment, along with experiment N14 at SEQUOIA, will use a direct geometry time of flight spectrometer to measure excitations (in our case, spin waves). Along with N2, N7, N12, N13 and N17 we will observe powder rings, Bragg peaks from polycrystalline samples. Like N10 using the Magnetism Reflectometer, we will not only measure magnetic scattering, but will also demonstrate how polarization analysis can distinguish between magnetic and other types of scattering.

N17: SNAP Spallation Neutrons at Pressure, SNS BL3

Pressure-induced phase transitions of water at room temperature

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.

N18: NSE Neutron Spin Echo Spectrometer, SNS BL15

Dynamics of Surfactant Micelles

We will investigate the dynamics of sodium dodecyl sulfate (SDS) micelles. The goal of the experiment is to measure the effective diffusion coefficient of the SDS micelles suspended in heavy water. This "classic" NSE experiment will allow us to illustrate the basic principles of the NSE technique and the required measurements and corrections. We will go through the reduction process starting from raw data to the intermediate scattering function. Finally, by comparing the results with model calculations, we will show the link between the structure and the dynamics in colloidal fluids.

2017 X-ray Experiment descriptions:

X1: High Energy X-ray Scattering for Strain Measurement, 1-ID

"Measuring material response using in-situ high energy diffraction microscopy and powder diffraction techniques"

Jun-Sang Park, Hemant Sharma, and Jonathan Almer

Polycrystalline materials encompass large groups of materials such as metals, ceramics, and minerals are utilized 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 material response to loading using high energy diffraction microscopy (individual grain response) and powder diffraction (aggregate response).

X2: X-ray Tomography, 2-BM

"Effects of propagation phase contrast imaging low contrast samples "

Francesco De Carlo

Propagation phase contrast effect is a very powerful technique when imaging low contrast objects, in which the density of the different components is very similar. This is the case of most part of biological samples, such as soft tissues, embryos, medical samples and so on. In this experiment, we will evaluate the differences between using an absorption vs a propagation phase contrast protocol in biological materials (bone and soft tissue). We will show how x-ray tomography is performed including basic tomography principles, sample mounting and alignment, data collection, data analysis and 3D rendering.

X3: Nuclear Resonance Scattering, 3-ID

"An introduction to NRIXS"

Michael Hu and Ercan Alp

Nuclear Resonant Inelastic X-ray Scattering (NRIXS) is a spectroscopy method to study atomic vibrations and dynamics, currently done with synchrotron radiation at a few high energy third generation facilities. It finds a wide range of applications in condensed matter physics, materials science, chemistry, biophysics, geosciences, and high-pressure researches. 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. Many atomic dynamics and lattice thermodynamics information can be derived from NRIXS measurements. Phonon Density of States (DOS) characterizes lattice dynamics of a material and can be derived under the quasi-harmonic approximation. Combined with modeling and simulations, results from NRIXS can provide unique and clarifying insights into many fields of research.

X4: X-ray absorption spectroscopy measurements at 5-BM-D

Denis Keane and Qing Ma

X-ray absorption spectroscopy techniques have been widely used in the research activities of multiple disciplines, for example chemistry, chemical engineering and environmental science. These techniques are also very versatile and can be adapted to suit a variety of sample conditions, including bulk, thin film, powder, and liquid. Measurements can be carried out through various channels or modes such as absorption, fluorescence, electron yields, etc., and in various geometries from normal incidence geometry (for transmission or grazing exit fluorescence measurements) to grazing incidence geometry. We will demonstrate elemental selectivity and chemical speciation in several types of samples including bulk mixtures and thin films.

X5: Energy Dispersive X-ray Diffraction, 6-BM

"Energy Dispersive X-ray Diffraction"

John Okasinski

The energy-dispersive x-ray diffraction (EDXRD) configuration enables one to selectively measure scattering from a discrete 3D volume within a larger bulk sample and surrounding environment. This is achieved through the use of a polychromatic incident beam and measuring at a fixed scattering angle with an energy-dispersive detector. The gauge volume attained creates the opportunity to map both crystalline phases and strain in complex samples. Three examples that make use of this technique include: mapping the progress and heterogeneity of the electrochemistry within a battery; mapping the strain in a structural component, such as near a weld joint; a sample confined inside of a complex environment, such as a furnace or large volume, high pressure cell.

X6: Magnetic X-ray Scattering, 6-ID-B

"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 and in external fields.

X7: Radiography, 7-BM

"Time resolved radiography of liquid fuel sprays"

Alan Kastergren

Multiphase flows are critical to numerous technologies we depend upon. For example, liquid sprays have a large impact on the performance of internal combustion engines and liquid rockets. These flows are typically opaque to visible light, which makes measurements of these flows challenging. Time-resolved x-ray radiography has been developed over the past 15 years at Argonne to quantitatively probe these flows, and now represents one of the best ways to study dense multiphase flows. Students will use radiography to probe the structure of a commonly used spray flowfield as a function of space and time.

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

"Time-resolved x-ray diffraction"

Don Walko, Anthony DiChiara, Haidan Wen

Heat transport is becoming an increasingly critical issue in the design of nanoscale electronics; as device components shrink, the interfaces between components play a more prominent role. In this experiment, laser-pump, x-ray diffraction-probe measurements will be used to determine the thermal conductance of a film/substrate interface. Pulses from an ultrafast Ti:sapphire laser will be used to heat a thin metal film grown on a transparent substrate. X-ray Bragg diffraction then probes 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 angular shift of the film's Bragg peak is thus mapped out for several laser/x-ray time delays. The position of the Bragg peak acts as a thermometer for the film, from which the conductance of the film/substrate interface will be derived.

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

"Auto forensics: XANES analysis of catalytic converters"

Tianpin Wu, George Sterbinsky, Steve Heald

All automobiles have catalytic converters, which are important for controlling emissions. All catalytic converters contain a catalyst. When a catalyst fails, it is rarely due to a problem with the converter. It is typically a symptom of something else. This experiment will demonstrate how spectroscopic techniques can be used to determine what materials are in a spent catalyst from a catalytic converter. This information can in turn be used to deduce what may have been wrong with the automobile engine. For a fragment of catalyst obtained from a local muffler shop, an energy-dispersive detector will be used to identify the elemental composition through the use of calibration foils. Then, the XANES of select elements will be obtained, and chemical fingerprinting will be used to identify the compounds. The students will be guided through the process of coming up with a hypothesis as to "what killed the car?"

X12: Ultra-Small Angle X-ray Scattering, 9-ID

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

Jan Ilavsky, Ross Andrews, Ivan Kuzmenko

This instrument provides a unique facility for ultra-small-angle, small-angle, and wide-angle scattering studies over an unprecedented range of length scales 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.

X13: Synchrotron Powder Diffraction, 11/17-BM

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

Saul Lapidus, Wenqian Xu & Andrey Yakovenko

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 strategies for data collection, processing, 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 detector measurements, as well as a wide range of in-situ sample environments. They will learn how to access and use these tools to address scientific challenges related to their own research. The second day of this experiment will include an interactive tutorial on data processing and Rietveld analysis using the software package GSAS-II, including the determination of 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, in depth questions will also be addressed if time permits.

X14: Pair Distribution Function, 11-ID-B

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

Olaf Borkiewicz, Kamila Wiaderek, 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.

X15: Small Angle X-ray Scattering, 12-ID-B

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

Xiaobing Zuo, Byeongdu Lee

Small angle X-ray scattering (SAXS) and Grazing incidence SAXS (GISAXS) are the scattering techniques to determine nanoscale structures and provided at 12-ID-B stations of APS. Examples of research experiments performed at the beamline include in-situ nanoparticle growth, in-situ monitoring nanoparticle catalyst under reaction, block copolymer morphology, aggregation of charged polymers, self or directed assembly of nanoparticles, structure of gel, conformation of protein and RNA, nano and bio hybrid materials, and so on. In this experiment, the beamline and its capabilities will be introduced, and measurements will be carried out on a variety of different samples, i.e., proteins or polymers or nano-particles or nano-particle assemblies. The data will be analyzed and interpreted.

X16: 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.

X17: X-ray liquid surface scattering, 15-ID-C

“Biomolecules at air-water interface”

Binhua Lin, Mati Meron and Wei Bu

Many biochemical processes and reactions occur at liquid surfaces and interfaces. These include interactions between cells and the extracellular matrix, protein interactions at cell and organelle membranes, gas transfer at the lung tissue-air interface, and drug intake by cell membranes. Synchrotron x-ray surface scattering techniques are used to determine structure on the subnanometer length scale at soft, hydrated interfaces of biological interest. The goal of this experiment is to determine the structure and ordering of a Langmuir monolayer of phospholipid molecules, Dipalmitoylphosphatidylcholine (DPPC), which is the major constituent of lung surfactant (a Langmuir monolayer consists of a single layer of amphiphilic molecules supported at the air-water interface).

We use Langmuir trough method to prepare the monolayer of DPPC at the surface of water. X-ray reflectivity (XR) techniques will be used to measure the electron density profile (or structure of the monolayer) normal to the surface of water, and grazing incident x-ray diffraction (GIXD) will be used to measure the packing of the lipid molecules along the water surface. Results of those measurements will then be analyzed through model fitting routines to determine the molecular structure and packing of the lipids at the surface of water.

X18: 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.

X19: Fundamentals of beamline operation, 20-BM

"Fundamentals of beamline operation and Cu XAFS"

Steve Heald and Chugjun Sun

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: Grazing Incidence Interface Diffraction, 33-BM

"Exploring 3-D Reciprocal Space: a Powerful Tool to Answer Basic & Applied Materials Science Questions"

Evguenia Karapetrova

The efficient exploration of large volumes of reciprocal space, made possible by the advent of high frame rate and low noise x-ray area detectors, allows for rapid characterization of a sample's structure and morphology, as all of its crystalline phases and their orientations can be determined simultaneously. The method is particularly powerful if not all the constituent phases (and the corresponding locations of their diffraction signals) are known, and aids in the discovery of unexpected phenomena or crystal structures.

X21: Coherent X-ray Diffraction Imaging, 34-ID-C

"Coherent X-ray Diffraction Imaging of Nanocrystals"

Ross Harder

The high brightness, and resulting high degree of coherence, of modern synchrotron x-ray sources has enabled the development of advanced x-ray imaging techniques. Coherent x-ray diffraction (CXD) imaging exploits the coherence of the synchrotron source to replace the lens of a traditional microscope with computational algorithms to form images. This imaging method allows one to surpass the resolution limits of modern x-ray optics. It also provides for an unencumbered space around the sample for complex in-situ environments. In addition, when the coherent scattering in the vicinity of a Bragg peak of a crystal is measured, a high sensitivity to distortions of the crystal lattice due to strain can be exploited. In this experiment we will measure the coherent scattering in the vicinity of a Bragg peak of a small (typically 300nm) gold crystal. We will then computationally invert the measured 3D diffraction pattern to a 3D image of the crystal.

X22: 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.